











## **THE SOAP MAKER'S HAND BOOK**





# THE SOAP MAKER'S HAND BOOK

MATERIALS, PROCESSES, AND RECEIPTS FOR  
EVERY DESCRIPTION OF SOAP

INCLUDING

FATS, FAT OILS, AND FATTY ACIDS; EXAMINATION OF FATS AND OILS;  
ALKALIES; TESTING SODA AND POTASH; MACHINES AND UTENSILS;  
HARD SOAPS; SOFT SOAPS; TEXTILE SOAPS; WASHING POWDERS;  
AND ALLIED PRODUCTS; TOILET SOAPS, MEDICATED SOAPS,  
AND SOAP SPECIALTIES; ESSENTIAL OILS AND OTHERS;  
PERFUMING MATERIALS; TESTING SOAPS.

EDITED CHIEFLY FROM THE GERMAN OF  
**DR. C. DEITE, A. ENGELHARDT, F. WILTNER,**  
AND NUMEROUS OTHER EXPERTS.

WITH ADDITIONS

BY

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## PREFACE TO THE SECOND EDITION.

THE first edition of this work appeared under the title A PRACTICAL TREATISE ON THE MANUFACTURE OF SOAP AND CANDLES. It has been out of print for some years, but nevertheless there is a constant demand for it, and this together with the fact that frequent inquiries are received for information in this important department of industry are the inducements which have led to the preparation of the present treatise.

Since the appearance of the first edition much progress has been made in the manufacture of soap, and in the present volume it has been endeavored to place before those interested in this industry a practical and comprehensive account of modern methods of operation.

The portion of the volume devoted to the manufacture of the various kinds of soap has been almost entirely rewritten. Old methods of working have been eliminated and new processes introduced, particular attention having been paid to the working of fatty acids with alkaline carbonates—the so-called carbonate-saponification.

In addition to exhaustive directions for the manufacture of all kinds of soap, both by boiling and the cold and semi-warm processes, numerous formulas of stocks available for the purpose are given, as well as receipts for washing powders, liquid soaps, medicated soaps and other soap specialties.

In order to make the present treatise as complete and reliable as possible, much time and care has been devoted to the gathering of information from all available and widely-scattered sources. The book is, however, chiefly based upon three German works, viz.: *Handbuch der Seifenfabrikation*,

edited by Dr. C. Deite; *Handbuch der praktischen Seifenfabrikation*, by Alwin Engelhardt, and *Die Seifenfabrikation*, by F: Wiltner. These works are the results of many years of practical experience, and are by men of acknowledged authority.

As is their long-established practice, the publishers have provided the volume with a full table of contents and an exhaustive index, thus making reference to any subject in it both prompt and easy.

W. T. B.

PHILADELPHIA, April, 1912.

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## CHAPTER I.

### HISTORICAL REVIEW OF THE MANUFACTURE OF SOAP.

It is frequently asserted that soap was known to the authors of the Old Testament; but the Hebrew words used in the passages<sup>\*</sup> are stated by authorities to refer to vegetable and mineral lyes, i. e. potash and soda in some form.

In Homer's time the cleansing of clothes seems to have been effected by simply rubbing or pounding in water without any addition, for he tells how Nausicaa and her attendants washed clothes by stamping them with their feet in pits filled with water.

Later on the juices of certain plants were employed as detergents, and also natural soda and wood ashes, and the fact that the strength of alkalies can be increased by lime was already known to Paulus Aegina. Fuller's earth was, however, the principal agent used for washing in ancient times, the fuller's art being due, it appears, to one Nicetus, the son of Hermaeus. The Roman fullers (*fullones*), who washed dirty garments, were persons of no little importance. Their trade and the manner of carrying it on were regulated by laws. At one time, fuller's earth, found of a superior quality in Staffordshire, and other counties of England, was considered so indispensable for the dressing of cloth that, to prevent foreigners from rivaling English fabrics, it was made a contraband commodity, and its exportation made equally criminal with the heinous and wicked export of wool.

The elder Pliny gives us the earliest account of soap as having been first manufactured by the Gauls, and used by

\* Jeremiah II., 22, and Malachi III., 2.

(1)

them as a cosmetic, and for dyeing the hair red. He also states that it was made from tallow and ashes, the best being prepared from goat's suet and beechwood ashes, and that the Gauls employed it both in a solid and liquid form.

From this statement by Pliny it has been generally concluded that the invention of soap was due either to the Gauls or the Germans. E. Moride,\* however, contests the correctness of this conclusion. He is of the opinion that Pliny's statement simply refers to the application of soap as a cosmetic and hair-dye, and believes the Phoenicians, who settled in Gaul 600 B. C., to have been the actual inventors of soap.

It cannot, however, be supposed that the first soap was an artificial product like that of the present day. It was very likely a mixture of oil and wood ash, which was used as a salve in eruptions of the skin and similar diseases. Later on it may have been accidentally discovered that a more effective salve was obtained by mixing the ash with water and burnt lime before combining it with the oil. Thus, no doubt, products were gradually obtained which resembled the present soaps of Algiers, of which Leon Droux writes:† "In the interior of Algiers the Cabyles bring to market a mass which serves the double purpose of a remedy and for household use. It is a soap prepared almost in the cold way, of a slightly yellowish color, somewhat transparent, and of a jelly-like consistency, but with a very small content of water. It is made from olive-oil and lye, the latter being prepared by allowing water to percolate through a mixture of wood ashes and burnt lime. The Arabs use the salve-like product thus obtained for afflictions of the skin, as well as for household purposes, and for washing wool to be worked into tissues."

As a detergent, soap is first mentioned by the authors of the second century after Christ. The celebrated physician, Galenus, speaks of it as a detergent, as well as a medicament,

\* *Les Corps gras industrielles.*

† *Les Produits Chimiques, Paris, 1878, p. 186.*

and considers the German soap as the best, and the Gallic as the next best.

But little is known of the further gradual development of the soap industry. Marseilles, it is said, carried on a considerable trade in soap as far back as the ninth century. In the fifteenth century, Venice was the principal market, but was out-stripped in the seventeenth century by Savona,\* Genoa, and Marseilles.

It is somewhat uncertain when soap making was first introduced into England, but it was probably in the fourteenth century. The first patent for improvements in the manufacture of soap was obtained, in 1622, by Messrs. Jones and Palmer. At the same time a company obtained a monopoly for the manufacture of soap, paying annually a tax of £20,000 for 3000 tons of soap.

In France the monopoly system was also in force in the seventeenth century. In 1660, Pierre Rigat, a merchant of Lyons, made a proposition to the King to manufacture by special methods sufficient soap for the needs of France without importing any of the materials required in the manufacture. Louis XIV. accepted the proposition and granted him the sole privilege for twenty years of erecting factories for the manufacture of white, mottled, and all other kinds of soap in any location it suited him. The six or seven factories then in existence were allowed to remain, but under the condition that they should not increase their capacity and would sell their products to Rigat at a fixed price. This patent, being the cause of many disputes, was revoked in 1669.

But little is known about the soap industry in Germany during the early centuries. The business was carried on on a small scale, and this could not well be otherwise, since, with the impure raw materials, principally crude tallow and wood ashes, one boiling frequently required as many days as hours at present. The industry was further hindered by the general

\* Whence the French name for soap (*savon*).

practice of every household preparing its own soap, which continued up to the introduction of artificial soda and tropical vegetable fats. But since then many large factories have been established, and, as the German soap-boilers applied to the trade its true chemical character, they produced superior goods. The soft soap of Germany is still much used for household purposes as well as for manufacturing, and it has acquired a reputation for excelling in quality that of other countries.

In this country there has been a steady progress in the improvements constantly making in this important branch of industry, until now we are producing goods which for quality compare favorably with any made elsewhere; moreover, we have invented much new and improved machinery and apparatus that greatly facilitate the processes, saving labor and time and improving the quality. Thus the United States is at this time but little behind any other country, either in the amount made or in the quality of the article; while in the economy and facility of their manufacture this industry is fully abreast, if not in advance, of that of nearly all other countries, and is steadily progressing, so that it cannot be long before we shall equal in quality and excel in quantity.

Not much advance was made in the manufacture of soap until at the beginning of the present century it commenced to attract the attention of scientific men, and Leblanc gave to the world his splendid process for the production of soda from common salt. But, like many other benefactors of the human race, Leblanc did not reap the reward for his invention. In 1791 a patent for fifteen years was granted to him in France, which was, however, shortly after revoked. The manufactory, which Leblanc had established by the aid of the Duke of Orleans, was just beginning to work when the Revolution put an end to all business; the property of the Duke of Orleans was seized, and the factory being included, the fabrication was stopped. Soon, the Continental wars preventing the importation of Spanish sodas, the French industry felt the loss

of this important element so essential to its work.' Then, on the proposition of Carnot, the Committee of Public Safety obliged Leblanc to sacrifice to the country the fruit of his discovery by making it public. Leblanc was ruined, and after many struggles for redress fell a victim to melancholy, and died, in 1806, by his own hand and in abject poverty.

The next great discovery, and not second in importance, was due to Chevreul, who, by describing the exact constituents of the fatty bodies and making known the processes for their separation, raised soap-making from empiricism and guess-work to its present position as a truly scientific art. By establishing the practical and scientific basis upon which the soap industry is now carried on, Chevreul and Leblanc may be considered as its founders.

The introduction of new fats and oils, especially of palm oil, palm-kernel oil, and cocoanut oil, added an important variety to the list of soaps, particularly of toilet soaps. The employment of caustic soda, which enables the soap boiler to prepare with great ease caustic lyes of a high degree, was also of great importance, as well as the production of potash from calcium chloride, and that of caustic soda in the electrolytic way.

A considerable change in the mode of boiling appears to have been brought about by the use of fatty acids for the manufacture of soap, and the saponification by alkaline carbonates connected therewith. When about fifty years ago, the price of glycerin rose enormously, many factories engaged in the business of abstracting the glycerin from the fats and selling the fatty acids to the soap manufacturer. Some larger soap plants also installed at that time autoclaves for the saponification of fats, and the question has recently been again agitated, particularly by the introduction of fermentative splitting of fat, chiefly because the latter requires a less expensive plant than autoclave saponification.

Material changes and improvements have been made in the machinery used in the manufacture of soap.

The primitive system of slabbing by hand by drawing a wire, in parallel lines of uniform distance apart, through the frame of soap, has been almost entirely replaced by mechanical slabbing devices. Mechanical crutchers have been largely substituted for hand crutchers, thus doing away with the laborious work connected with the latter. Great improvements have also been made in presses, this being of importance not only for toilet soaps, but also for domestic soaps, as more value is now attached to the external appearance of the latter than was formerly the case. Iron frames are now largely used in place of wooden ones. To prevent the soap from cooling too rapidly, the frame is sometimes enveloped in a bad conductor of heat in the form of a mattress stuffed with tow.

In modern times it has been endeavored to shorten the time for the production of soap by avoiding long cooling in the frame. Pioneers in this respect were A. & E. des Cressonières, of Brussels with their *broyeuse sécheuse continue*. While only dried soap could formerly be used in the milling machine, the above-mentioned apparatus allows of the working of the hot liquid soap as it comes from the kettle, and soap finished in the kettle yesterday can, perfumed and colored, be brought the next day into commerce. This invention was originally intended only for the preparation of milled toilet soaps, but it was soon found to be of use also for the manufacture of various kinds of domestic soap, and the idea has been utilized in the construction of soap-cooling machines.

The manufacture of soap in a closed boiler under pressure has been frequently tried. Thus, Arthur Dunn, a number of years ago, patented in England a process, the object of which was to accelerate the process of saponification by effecting it in a pressure-boiler at a temperature of from 302° to 320° F. Mouveau later on proposed to effect the manufacture of soap in a pressure-boiler provided with a safety-valve. The boiler was furnished with a stirrer and was surrounded by a jacket into which steam for heating the boiler, or water for cooling

could be introduced. A temperature of 302° to 320° F. was produced in the boiler. Previous to boiling, the fat and lye were introduced through a man-hole which was then closed; after the air had been expelled the safety-valve was also closed. Additions of fat or lye required after the commencement of boiling were forced into the boiler by means of a pump. The spent lye and finished soap were discharged through a pipe provided with a stop-cock, which passed from the bottom of the boiler through the jacket.

However, all these methods for the preparation of soap in pressure-boilers have been abandoned because they did not prove satisfactory, and there is no probability that the desired result will be attained by such experiments. Saponification is, to be sure, rapidly effected in a closed boiler under pressure, but the saponified fat is not soap such as is demanded by commerce. Besides the saponification of the fats, there are other operations in the production of the artificial product known as soap, and in order to be quite sure, these operations can only be carried on when the soap-boiler has the soap before him in the open kettle.

The manufacture of soap is at the present time connected with greater difficulties than it was thirty or forty years ago. The frequent changes in the prices of raw materials, while the prices of the soaps themselves have become very low, allow no longer of working by rule of thumb, and force the soap-boiler to adapt his manufacturing processes to prevailing conditions in order to enable him to compete with others.

## CHAPTER II.

### RAW MATERIALS USED IN THE MANUFACTURE OF SOAP.

THE ordinary soaps of commerce consist of the more or less impure sodium or potassium salts of the fatty acids. They are prepared by treating fats and fat oils, and also fatty acids, with lyes. The latter are obtained by treating aqueous solutions of alkaline carbonates—soda or potash—with quick lime, or by simply dissolving the caustic alkalies in water. For some soaps an addition of rosin is also used.

The raw materials employed in the manufacture of soap, therefore, consist, on the one hand, of fats, fat oils, fatty acids and rosin and, on the other, of alkalies. To these have to be added as auxiliary raw materials: Water, lime, and common salt. Besides the above-mentioned materials which are indispensable for the manufacture of soap, foreign substances are frequently incorporated with the soap, chiefly with the object of cheapening the cost of manufacture. These substances are known as filling materials, soda ash, silicate of soda (water glass or soluble glass), glue, starch, etc., being used.

It is scarcely necessary to mention that a thorough acquaintance with the properties of the raw materials entering into the manufacture of soap is of great importance, since on it depends not only the success of the operation itself, but, besides the quality, also the quantity, *i. e.* the yield of soap, the latter varying with different fats.

#### FATS.

*Nature of fats.*—In the soap industry animal as well as vegetable fats are employed. These fats possess the following common properties: They feel unctuous to the touch and form oleaginous fluids, either at the ordinary temperature or when heated. Dropped upon paper they form transparent spots

which do not vanish in time nor by the application of heat. They are specifically lighter than water and insoluble in it, but are soluble in ether, carbon disulphide, and volatile oils. They are not volatile and commence to boil at from 572° to 608° F., suffering decomposition thereby. By themselves they burn with difficulty, but when used with a wick, with a luminous flame.

According to their consistency, the fats are divided into solid fats or tallow, semi-solid fats or butters and lards, fluid fats or oils, and train oils, the latter term being applied to the fluid fats derived from various marine animals. The solid fats melt readily, most of them liquefying below 212° F. The liquid fats, *i. e.* the oils, solidify at a low temperature. At ordinary temperature the oils are not thinly-fluid like water, but are distinguished by a certain degree of viscosity which has to be taken into consideration when they are to be used as lubricants. Castor oil is the most viscous of all oils known at present.

Of all liquids, the fluid fats expand the most by heat. According to Preisser, the expansion of 1000 parts by volume amounts for 1° Centigrade, with olive oil to 0.83 part by volume, with rape oil to 0.89, and with train oil to 1.0, so that 1000 liters of olive oil measured in winter at 0° C., will measure in summer at 20° C. 1016.6 liters, rape oil measured in the same manner 1017.8 liters, and train oil 1020 liters.

The oils, when shaken with water, which has been made mucilaginous by dissolving albumen or gum-arabic in it, remain suspended in the form of microscopically small drops, and the fluid acquires a milky appearance; it forms an emulsion.

The fats, as generally found, vary in color, the solid fats being mostly white or yellowish, the oils, yellow or yellow-green, and the train oils, red or red-brown. They possess odor and taste by which they can be distinguished, for instance, the smell of tallow, the aromatic odor of palm oil and cocoanut oil, the taste of edible fats such as butter, olive oil etc., as compared with the nauseous taste of rape oil.

On exposure to the air most fats gradually undergo a change, some of them being converted into a solid, transparent mass—a varnish. Such oils are known as *drying oils*. They absorb oxygen from the air and pass into a higher state of oxidation. Others, called *non-drying oils*, never dry entirely, but only acquire a greater degree of consistency and turn rancid.

Many solid fats also become rancid, i. e. they suffer a kind of fermentation by which the fatty acids are set free and impart their odor and taste to the fat. Commercial palm oil and cocoanut oil are frequently in such a state of decomposition, but the bad odor is wanting. Rancidity, however does not affect the availability of the fats for the manufacture of soap, as the liberated fatty acids are neutralized by saponification with the alkali.

While pure unaltered fats, with the exception of castor oil, are next to insoluble in cold alcohol, when rancid, they dissolve readily in it and show an acid reaction. Acid reaction of fats, when not due to adhering foreign substances, always indicates commencement of rancidity, as in a pure unaltered state the fats are entirely neutral.

*Chemical constitution of fats.*—According to their elementary composition the fats consist of carbon, hydrogen, and oxygen, while mineral fats and oils consist only of carbon and hydrogen. The following table shows the elementary composition of some of the fats:

	Carbon. per cent.	Hydrogen. per cent.	Oxygen. per cent.
Cocoa-Butter . . . . .	75.20	11.90	12.90
German sesame-oil . . . . .	76.80	11.50	11.70
Lard . . . . .	74.54	11.94	11.26
Linseed-oil . . . . .	78.00	11.00	11.00
Mutton-suet . . . . .	78.10	11.70	9.30
Olive-oil . . . . .	77.21	13.36	9.43
Ox-tallow . . . . .	76.50	11.91	11.59
Poppyseed-oil . . . . .	76.63	11.63	11.74
Rape-oil . . . . .	77.21	13.36	9.43

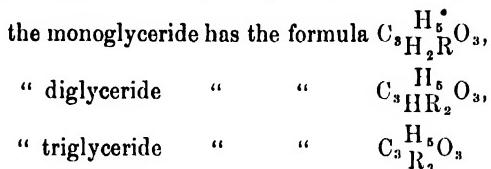
As shown by the above table, there is considerable variation in the elementary composition of the fats and, moreover, there can scarcely be any doubt that the fat from the same animal is not always alike in composition. This is shown by the more proximate composition of the fats. The latter, as they occur in nature, are not simple chemical combinations but mixtures of such.

An important step towards the investigation of the nature of fats was, in 1779, made by Scheele, who discovered, whilst engaged in the preparation of lead plaster, a sweet body soluble in water. He called it "*principium dulce oleorum*," and it is now generally known as glycerin.

However, the principal elucidation of the nature of fats owe to the labors of Chevieu, which commenced in 1812, and culminated, in 1823, in the publication of his celebrated work : "*Recherches sur les Corps gras d'origine animale*." His researches served as a basis for all further studies of the chemical behavior of the fats. The correctness of his observations was in the main confirmed, and his researches extended by others.

Nearly all the fats occurring in nature are peculiar combinations of glycerin, a body composed of 3 equivalents of carbon, 8 of hydrogen and 3 of oxygen, and are, therefore, termed *glycerides*. According to the older chemical view, the fats contained a body called glyceryl-oxide or lipyloxide, combined with 3 equivalents of fatty acids, and glycerin was designated as glyceryl-oxide, or as glyceryl-oxhydrate. But, according to modern chemistry, glycerin is a so-called triatomic alcohol. If, as stated above, glycerin contains 8 equivalents of hydrogen, 3 of them can be represented by acids. If we designate carbon C, hydrogen H, and oxygen O, glycerin has the formula :  $C_3H_8O_3$ , which can also be written :  $C_3H_8H_8O_3$ , or  $C_3\overset{H_8}{H_3}O_3$ . The 3 equivalents of hydrogen, written separately in the last two formulas, can be represented by acids. Now if 1 equivalent of hydrogen is replaced by an

acid, a so-called monoglyceride is obtained; if 2 equivalents are replaced, a diglyceride; and if all 3 equivalents are replaced, a triglyceride. By designating an acid R,



All glycerides occurring in nature, and others thus far examined, are triglycerides, *i. e.*, glycerin in which 3 equivalents of hydrogen are replaced by acids. The acids contained in fats are generally termed fatty acids, the most important being stearic, palmitic, oleic, and linoleic acids.

Many train oils contain ether-like combinations of another alcohol—cetyl alcohol ( $C_{10}H_{22}O$ )—and wool fat contains ether of cholesterol, as well as that alcohol itself.

**Fatty acids.**—The fatty acids, which can be separated from the fats, may, according to their composition, be divided into four groups: Into acids, 1, of the composition  $C_nO_{2n}O_2$  (usually called acetic series of acids); 2, of the composition  $C_nH_{2n-2}O_2$  (acrylic or oleic acid series); 3, of the composition  $C_nH_{2n-4}O_2$ ; and 4, of the composition  $C_nH_{2n-2}O_3$ .

The fatty acids of the first group are called *saturated* fatty acids, and the rest *unsaturated* fatty acids.

In the following table the naturally-formed fatty acids are given together with their occurrence, properties and chemical composition:

Name of acid.	Occurrence.	Properties.	Chemical composition
Palmitic acid.	Palm oil	crystallizes in needles; melts at 143.5° F.; odorless and tasteless; reddens litmus paper.	$C_{16}H_{32}O_2$

Stearic acid.	Animal fats	crystallizes in laminae; melts at 156° F.; odorless and tasteless; reddens litmus paper; distills without alteration.	C <sub>18</sub> H <sub>36</sub> O <sub>2</sub>
Margaric acid.	Animal fats	is by many considered a mixture of palmitic and stearic acids; melts at 140° F.	C <sub>18</sub> H <sub>36</sub> O <sub>2</sub>
Butyric acid.	Gastric juice; butter; perspiration	an oily liquid; colorless; smells of rancid butter; caustic, reacts and tastes sour; boils without alteration at 312.5° F.	C <sub>4</sub> H <sub>8</sub> O <sub>2</sub>
Lauric acid.	Cocoanut oil	crystallizes in silky needles; melts at 109.4° to 113° F.; acid reaction.	C <sub>10</sub> H <sub>20</sub> O <sub>2</sub>
Myristic acid.	Cocoanut oil; spermaceti, nutmeg-butter	snow-white crystalline mass; melts at 127.4° F.; acid reaction.	C <sub>14</sub> H <sub>28</sub> O <sub>2</sub>
Oleic acid.	Non-drying oils	an oily liquid; colorless, tasteless and odorless; shows no acid reaction; congeals at 39.2° F.; absorbs oxygen with avidity.	C <sub>18</sub> H <sub>34</sub> O <sub>2</sub>
Ricinoleic acid.	Castor oil	an oily liquid; odorless; acid taste; shows an acid reaction when cooled to 32° F.	C <sub>18</sub> H <sub>34</sub> O <sub>2</sub>
Physetoleic acid.	Train oils; spermaceti	colorless and tasteless acicular mass; melts at 92.3° F.; becomes reddish on exposure to the air and then has a strong rancid odor.	C <sub>18</sub> H <sub>36</sub> O <sub>2</sub>
Brucic acid.	White and black mustard seed oil	crystallizes in laminae and needles; melts at 93.2° F.	C <sub>18</sub> H <sub>34</sub> O <sub>2</sub>
Linoleic acid.	Non-drying oils	an oily thinly-fluid liquid; slightly yellow; slightly acid reaction; refracts light; absorbs oxygen with avidity, becoming thereby tough and thickly fluid.	C <sub>18</sub> H <sub>36</sub> O <sub>2</sub>

The salts of stearic acid and of the other non-volatile fatty acids are called soaps. The alkaline salts are soluble in boiling water, but in cold water not without decomposition; nearly all the other salts are insoluble in water or soluble with difficulty. By boiling stearic acid with watery solutions of carbonate of potash or soda, carbonic acid is expelled and stearates are formed. In their purest state the alkaline salts are crystallized. Their behavior in water is also characteristic of the alkaline soaps of other fatty acids, they being sparingly soluble in cold water, but when boiled with not too large a quantity of it, they yield a clear solution which, on cooling, congeals to a turbid viscid mass. With a large quantity of water, they do not yield a clear solution but a turbid fluid which, on shaking, gives a tenacious lather which remains standing for quite a long time. The stearates are separated from their solutions by common salt. By repeated treatment with common salt the potassium salt can be completely converted into the sodium salt. The alkaline stearates are readily taken up in the heat by alcohol; on cooling the soaps generally separate from concentrated solutions in a gelatinized state, but after long standing they assume a crystalline form. In ether and benzine they are insoluble.

*Potassium stearate* ( $C_{18}H_{36}O_2K$ ) forms crystals with a greasy luster which dissolve in 6.6 parts of boiling alcohol. By compounding its hot aqueous solution with a large volume of water, acid potassium stearate ( $C_{18}H_{35}KO_2.C_{18}H_{36}O_2$ ) in laminae with a nacreous luster, insoluble in water, is precipitated. *Sodium stearate* closely resembles the potassium salt; it consists of lustrous laminae. The *stearates of calcium, strontium and barium* form crystalline precipitates. The *magnesium* salt also precipitates in a crystalline form. It is soluble in hot alcohol sufficiently to allow of its recrystallization from it, but is insoluble in cold alcohol. The salts of the heavy metals, such as the salts of silver, copper and lead are mostly amorphous; the lead salt is fusible without decomposition at  $257^{\circ} F.$

Chevreul distinguished, among the fatty acids obtained by the saponification of fats, one which he termed margaric acid, which, however, has been shown by Heintz to be a mixture of stearic and palmitic acids.

For the manufacture of stearin candles the behavior of stearic and palmitic acids, when melted together, is of great importance. Heintz found that mixtures of fatty acids are analogous to many alloys of metals in showing a lower melting-point than their separate components. The following table shows the behavior of mixtures of stearic and palmitic acids:

Melting-point, Degrees F.	Composition of mixtures.		Manner of congealing.
	Stearic acid.	Palmitic acid.	
153.0°	90	10	Crystalline scales.
149.5	80	20	Slender crystalline needles.
145.0	70	30	" " "
140.0	10	90	Beautiful large needles.
135.5	20	80	Very indistinctly needly.
134.0	50	50	Large-foliated crystalline.
133.0	40	60	" " "
132.0	35	65	Non-crystalline, lustrous.
131.0	32.5	67.5	" " "
131.0	30	70	lusterless.

Though the lowering of the melting-points decreases the value of the mixtures for the manufacture of candles, this disadvantage is more than counterbalanced by the importance of the other alterations the fatty acids undergo by being melted together. The pure acids are soft, of a loose structure, and very friable. By mixing them together they acquire sufficient density and hardness, so that they can be subjected to the pressure required to expel the oleic acid. The pure acids in congealing contract to such a degree that candles prepared from them have an unsightly appearance. A mixture of the acids being but slightly crystalline to amorphous, candles manufactured from the semi-congealed mass are dense

and non-crystalline. Candles from pure fatty acids are soft, opaque, friable, and lustreless; candles from a mixture, hard, lustrous, and transparent.

The salts of oleic acid behave, as regards their solubility in water, in a manner similar to that of the solid fatty acids, the alkaline salts alone being soluble. On the other hand, all the other salts are soluble in alcohol and a few also in ether, the lead salt belonging to the latter. The silver salt of oleic acid is insoluble in ether. By the addition of an excess of alkali, sodium chloride, etc., the alkaline salts separate from their aqueous solutions. All the salts of oleic acid are softer than those of the solid fatty acids, and are mostly fusible without decomposition.

Sodium oleate ( $C_{18}H_{33}NaO_2$ ) can be obtained by crystallization from absolute alcohol. It dissolves in 10 parts of water at  $53.5^{\circ}$  F., in 20.6 parts of alcohol of 0.821 specific gravity at  $55^{\circ}$  F., and in 100 parts of boiling ether. The potassium salt forms a transparent jelly by far more freely soluble in water, alcohol, and ether than the sodium salt. The barium salt is a crystalline powder insoluble in water, and at  $212^{\circ}$  F. cakes without melting. By boiling alcohol it is taken up with difficulty. Lead oleate is a loose, white powder, melting at  $176^{\circ}$  F. to a yellowish fluid; after cooling it is, however, rigid and brittle, and remains transparent.

In the examination of fats, titrations of the free fatty acids are frequently executed. The best indicator for the purpose is phenolphthalein. The solution is prepared by dissolving 0.5 to 1 grammé of phenolphthalein in 1 liter of alcohol. It has a yellowish color, and is reddened by the slightest addition of alkali in consequence of the formation of the corresponding salts. These salts are completely decomposed by weak acids, so that fatty acids in alcoholic solution can be sharply titrated with phenolphthalein. Ammonia does not give a perceptible coloration with phenolphthalein and is therefore unsuitable for the titration of fatty acid. It must also be borne in mind that phenolphthalein salt is decom-

posed by carbonic acid, as well as that the bicarbonates of alkalies have not the same effect as the carbonates. Any carbonic acid present must, therefore, be removed by boiling.

*Alcohols of the fatty group.* The following alcohols take part in the constitution of fats: Glycerin, cetyl alcohol, cholesterolin, phytosterin, and a few others. The most important of these is

*Glycerin* ( $C_3H_8O_3$ ). In the pure state it is a sweet colorless liquid, specific gravity 1.262, of great viscosity, and miscible in all proportions with water and alcohol. It dissolves many substances, *e. g.* metallic salts, which are soluble in water. It takes fire at  $302^{\circ}$  F., and burns with a blue flame. A characteristic property of glycerin which may serve for its identification, is that of yielding an exceedingly pungent and irritating substance, known as *acrolein*, or *acrylic aldehyde*, when sharply heated, or subjected to the action of dehydrating agents.

*Cetyl alcohol* ( $C_{16}H_{34}O$ ), or *ethal* is a white crystalline mass without taste and odor. It melts at  $122^{\circ}$  F. and boils at  $651^{\circ}$  F. It is insoluble in water, soluble in alcohol, and very readily so in ether and benzine. It is a constituent of sperm-aceti oil.

*Cholesterin* ( $C_{28}H_{44}O$ ) crystallizes from chloroform in anhydrous needles of 1.067 specific gravity, which melt at  $293^{\circ}$  to  $295^{\circ}$  F. Cholesterin is insoluble in water and very sparingly soluble in cold dilute alcohol. It dissolves in 9 parts boiling alcohol of 0.84 specific gravity and in 5.55 parts of 0.82 specific gravity. It is readily taken up by carbon disulphide, chloroform, and benzine.

By carefully evaporating to dryness upon a crucible-lid a trace of cholesterin with a drop of concentrated nitric acid a yellow stain is obtained. On pouring ammonia over it this yellow stain assumes a yellow-red coloration. If a sample of cholesterin be triturated upon a crucible-lid with a drop of a mixture of 3 volumes concentrated hydrochloric acid and 1 volume ferric chloride solution, and carefully evaporated to

dryness, the particles remaining undissolved acquire a violet red, passing into a bluish, color.

Cholesterin is an essential constituent of wool-fat. Since all animal fats and oils contain small quantities of cholesterin, its presence in pure fat and oil indicates that the latter is of animal origin.

*Glycerides.* By far the most important glycerides are tri-stearin, tripalmitin and triolein. They are most widely distributed, most fats occurring in nature being mixtures of them. To the soap manufacturer the glyceride of lauric acid, or trilaurin, or laurostearin, is also of special importance.

*Tristearin* [ $C_{18}H_{36}(C_{18}H_{34}O_2)_3$ ], briefly called stearin, forms colorless nacreous scales which melt at  $161^{\circ}$  F., and congeal at  $158^{\circ}$  F. to an indistinctly crystalline mass. By heating stearin, however, to  $7^{\circ}$  above its melting-point, it congeals at  $125.5^{\circ}$  F. to a waxy mass, and then melts at  $131^{\circ}$  F. But by again heating it a few degrees above the latter melting-point it reassumes its original melting-point at  $161^{\circ}$  F. Stearin is insoluble in water, sparingly soluble in cold alcohol and ether, but freely in warm ether and boiling alcohol. It is the preponderating constituent of the tallows.

*Tripalmitin* [ $C_{16}H_{36}(C_{16}H_{34}O_2)_3$ ], or briefly palmitin, is contained in preponderating quantity in the lardaceous fats. It is best obtained pure from palm oil. It consists of small nacreous crystals, very sparingly soluble in cold alcohol, and somewhat more freely in boiling alcohol; on cooling it re-separates, however, in flakes. In boiling ether it is soluble in all proportions. When heated its behavior is very remarkable: it melts at  $123^{\circ}$  F., but on further heating re-congeals, and then only melts again at  $152^{\circ}$  F.

*Laurostearin* [ $C_{12}H_{24}(C_{12}H_{22}O_2)_3$ ] forms slender white needles grouped in the form of a star or tree. They melt at from  $111^{\circ}$  to  $115^{\circ}$  F., and congeal at  $73.5^{\circ}$  F. They are insoluble in water, very sparingly soluble in cold alcohol, but more readily in boiling alcohol, and freely in ether. Laurostearin is readily saponified by potash lye, forming a clear soap.

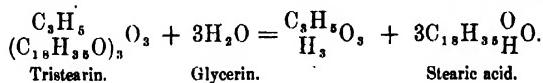
paste. When heated above its melting-point it is decomposed into acrolein and a solid body crystallizable from ether and alcohol.

*Triolein* [ $C_3H_5(C_{18}H_{35}O_2)_3$ ], briefly olein, forms the preponderating constituent of non-drying oils. In a pure state it is a colorless and odorless oil which crystallizes in needles at  $23^{\circ}$  F. It is insoluble in water, dissolves with difficulty in cold alcohol, but more freely in hot alcohol, and is miscible in all proportions with ether. Exposed to the air, it darkens and becomes acid and rancid gradually undergoing decomposition—*elaidin*. The latter consists of wart-like crystals which, according to Meyer, fuse at  $89.5^{\circ}$  F. and, according to Duffy, at  $100.5^{\circ}$  F. They dissolve very sparingly in alcohol, but readily in ether.

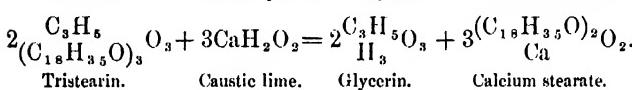
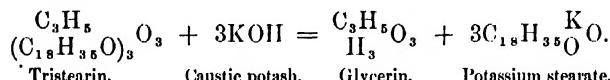
The combination resembling olein which is contained in drying oils, has been termed *olanin*, but its properties have been but little studied. Olanin does not become solid with nitrous acid.

Regarding the proportional quantities of stearin, palmitin, and olein occurring in the fats, it may be said that a fat is the more solid the more it contains of the first two, and the softer the greater the preponderance of the latter.

*Saponification of fats.*—By saponification was originally understood only the chemical process which takes place in boiling fats with strong bases whereby glycerin and fat acid salts are formed; but at the present time the term is applied to every reaction by which fats (even without the coöperation of bases) are resolved into glycerin and fatty acids. To understand the process of saponification we must consider that all glycerides are a variety of ethers, and as such possess the power of splitting by the absorption of water into their generators, *i. e.*, into glycerin and an acid. Therefore, for instance,



This splitting of the glycerides is called saponification. It already takes place with water alone, but only at a comparatively high temperature. It is facilitated by adding to the water a small quantity of a base or of an acid; but it is most readily accomplished by using together with the water a sufficient quantity of a base (potash, soda, or lime) to fix the acids formed, whereby the salts of these acids result, which are familiarly known as "soaps." The process is represented by the following equations:



In the laboratory the fats are most suitably decomposed with alcoholic potassium. Lewkowitsch gives for this the following directions: Heat in a porcelain dish upon a water bath, stirring constantly, 50 grammes of fat with 40 cubic centimeters of caustic potash solution of 1.4 specific gravity and 40 Cc. of strong alcohol until the soap becomes thick. It is then dissolved in 1000 Cc. of water, and the solution boiled over an open fire to expel the alcohol. This is best done by from time to time replacing the evaporated water. The soap is then decomposed by the addition of dilute sulphuric acid, and the solution heated so that the fatty acids settle as a clear oily mass upon the aqueous solution. The latter is then drawn off by means of a siphon, and the fatty acid several times washed with hot water until the sulphuric acid has been removed. The warm liquid fatty acids are filtered through a dry filter in a hot-water funnel.

With alkaline carbonates, the neutral fats can only be saponified at a higher temperature. Tilghman was the first who endeavored to technically utilize this fact. He mixed the melted fat with the quantity of alkaline carbonate solution

required for saponification and forced the mixture through a long coiled wrought-iron pipe of  $\frac{1}{2}$  inch interior, and 1 inch exterior, diameter which laid in the fire and was heated to from  $392^{\circ}$  to  $572^{\circ}$  F. Saponification commences already at  $383^{\circ}$  F.; it results more rapidly at a higher temperature. The mixture of fat and alkaline carbonate forced into the pipe comes out of the other end as soap. The liberated carbonic acid escapes through the same opening as the soap.

The splitting of the glycerides may also be effected by concentrated, or comparatively concentrated, sulphuric acid, whereby is first formed a sulphuric acid combination of the glycerin—sulphoglyceric acid—and perhaps also sulphuric acid combinations of the acids contained in the fats (sulpho-stearic acid, sulpholeic acid, etc.)

It may here be remarked that ammonia, the behavior of which is otherwise analogous to that of the alkalies, does not act in the same manner upon the fats. By shaking a fat oil with ammonia an emulsion is formed; by exposing this to the air the ammonia volatilizes in a short time, and the oil separates unaltered. The oil is also obtained unaltered when a quantity of dilute acid corresponding to that of ammonia is added to the mixture. This proves that ammonia and fat do not enter into combination by a simple mixture. However, by allowing ammonia to act upon fats in closed vessels, chemical combinations result, ammonia soap and the amide of the fatty acid being formed.

The utilization of the natural glycerides for the manufacture of soap and candles and the preparation of plasters, is based upon their splitting into fatty acids and glycerin. By plasters are generally understood the lead oxide salts of the fatty acids contained in the fats, though the term is also applied to combinations of these acids with other heavy metallic oxides obtained by precipitating soap solutions with metallic solutions.

*Technical methods of saponification.*—The soap boiler employs exclusively caustic alkalies for the saponification of

neutral fats. However, in the manufacture of stearin, the object of which is to obtain from the fats the solid fatty acids in a form which makes them suitable for candle material, various methods of saponification are used, such as saponifying with lime, with magnesia, with sulphuric acid and with water. The fats are first saponified in order to separate the glycerin, and then subjected to cold, and finally to warm, pressure.

The first attempt to utilize saponification for the preparation of a material suitable for the manufacture of candles was made by Gay-Lussac and Chevreul, who, in 1825, took out a patent for the separation of the fatty acids and their use in the manufacture of candles. The specifications of the patent are highly interesting, as they contain all the scientific principles which have up to the present day been applied to the manufacture of fatty acids, even saponification with acids which came into practical use only about 20 years later on. Nevertheless the patentees reaped no benefit from their invention. The history of this branch of industry furnishes a striking and instructive proof of the fact that there may be a long way between scientific truth and practical application. The processes chiefly recommended resembled too much the methods of the chemist in his laboratory and were entirely too complicated for technical practice. The alkalies served for saponification and, for the decomposition of the soap, hydrochloric acid the alkaline salts of which, as shown by experience, could never be completely eliminated from the separated acid, even by continuous washing with water, and the patentees even spoke of cold and hot alcohol for the complete purification of the fatty acids.

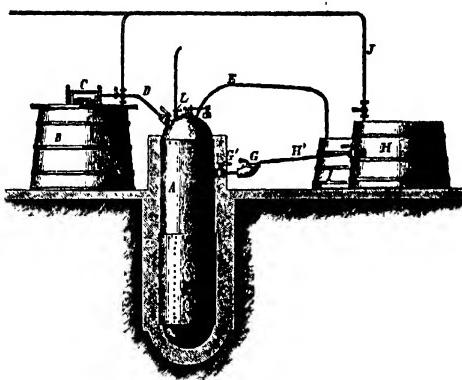
The manufacture of fatty acids was, in 1831, placed upon an economic basis by De Milly and Motard, who, in place of the alkalies, used lime for the decomposition of the fats, and decomposed the lime-soap formed by sulphuric acid. At first 15 per cent. of lime was used for the saponification of the tallow, but later on 8 to 9 per cent. was found to be sufficient

if the process is effected in covered pots and the mass thoroughly stirred during saponification.

The quantity of lime was later on still further reduced by a process given by De Milly. In this process a higher temperature has to be used and saponification is effected in a closed apparatus, called an autoclave. With the use of 2.5 parts of lime for 100 parts of tallow a temperature of 306° to 356° F.—8 atmospheres—is required.

Autoclave saponification is to-day carried on in essentially the same manner as recommended by De Milly. Fig. 1 shows

FIG. 1.



the apparatus used by him. *A* is a closed cylindrical vessel of copper, 4 feet in diameter and 16 feet high, and provided with a safety-valve, manometer, and manhole. By the pipe *L* it is connected with a boiler, which furnishes steam of a pressure of 8 atmospheres. The steam boiler consists of two heating tubes, placed above each other, and connected by two pipes. These two heating tubes have the same diameter ( $2\frac{1}{4}$  inches) and the same length (16 feet), and are heated by a fireplace.

Steam is introduced into the autoclave through the cock *L* and a pipe extending to the bottom. The pipes *D* and *E*, ex-

tending also to the bottom of the autoclave, serve for emptying the apparatus after saponification is finished.\*

The operation is carried on as follows: The charge of fat (4000 lbs.) is melted by steam in the wooden vat *H*, and conveyed through the funnel *G* into the apparatus; 220 gallons of milk of lime, containing 120 lbs. of lime, are then added. The apparatus being thus charged, the cock on the funnel, *G*, is closed, and the steam-cock *L* opened. The steam enters on the bottom, and, penetrating the milk of lime and fat, effects their mixture. The pressure in the autoclave soon rises to 8 atmospheres. In this manner the steam, which must always have the same pressure, is allowed to enter the apparatus for five or six hours, when it is shut off, and the apparatus allowed to rest for two hours for the water containing the glycerin to settle. The cock *D* is then opened, and, the pressure in the autoclave being still sufficient to force out the mass, the glycerin-liquor is first expelled, and passes through a filtering arrangement into the vat *B*. The glycerin having escaped and the fatty mass beginning to appear, the cock *D* is closed, the cock *E* opened, and the mixture of fatty acid and lime-soap brought into the vat *I*. The mixture being all in the vat, 240 pounds of sulphuric acid of 66° B., previously diluted to 14° or 15° B., are added with constant stirring and simultaneous introduction of steam, the decomposition of the lime-soap being finished in about two hours. The steam is then shut off and the fatty acids allowed to settle, when they are brought into another vat and washed.

Our knowledge of the interesting fact that water at a higher temperature is capable of decomposing fats is due to the investigations of Tilghman and Berthelot who, in 1854, almost simultaneously made this discovery, though Tilghman alone occupied himself with the industrial side of the question. According to his process, which, however, has everywhere

\*Instead of two discharge pipes, only one is generally used at the present time. It is provided outside of the autoclave with a three-way cock, from which connections lead to the glycerin vat and the fatty acid vat.

been found unsuitable for working on a large scale, the fat is brought in contact with hot water and is thus converted into a kind of emulsion. This mixture is driven by a forcing pump through a long wrought-iron tube which makes several bends in a furnace and is heated, according to the nature of the fat, to from  $500^{\circ}$  to  $660^{\circ}$  F. A treatment for ten minutes is claimed to be generally sufficient for the complete decomposition of the fats. Attempts were later on made to effect saponification in a closed apparatus—a digester—under high pressure. However by reason of the high pressure required for saponification this method presents great technical difficulties.

For the manufacture of stearin it is, of course, of great importance that saponification should be effected as completely as possible in order to obtain well-crystallizing fatty acids and a mass as free as possible from glycerin, so that in the subsequent pressing the separation of the solid, from the liquid, fatty acids is thoroughly effected. The soap manufacturer, on the other hand, aims to saponify the greatest portion of the fat in order to obtain as much glycerin as possible and it is of but little consequence to him whether a few per cent. of non-saponified fat remain in the fatty acids. Léon Droux is, therefore, of the opinion that in place of lime-saponification, which necessitates subsequent decomposition of the lime-soap formed by sulphuric acid, saponification can, in soap factories, be effected by water alone, and recommends for this purpose a closed apparatus. The latter is provided with a mechanical stirrer in the form of a copper shaft fitted with arms. By revolving this shaft the fat and water are thoroughly stirred and intimately mixed, the fat being thereby split into fatty acids and glycerin. Instead of saponification with water alone, it has been recommended to add a small quantity of caustic lye—containing about 2 per cent. of caustic soda—to the mixture of fat and water.

G. F. Wilson and G. Payne proposed the decomposition of the fat by superheated steam, and to distil over the acids

formed as well as the glycerin. However, the process did not come up to expectations on account of the great difficulty of keeping the temperature of the fat to be decomposed exactly at that required, namely,  $590^{\circ}$  to  $599^{\circ}$  F. If the temperature rises above the latter point two much glycerin is decomposed to acrolein, which renders the operation troublesome, and if it falls below the former, the process of decomposition progresses too slowly. If, however, the fat is exposed in a finely divided state to the superheated steam, the temperature can, according to Horschelt, be kept within much wider limits without more than the ordinary amount of acrolein being formed, or decomposition being retarded.

The behavior of fats towards sulphuric acid was first scientifically investigated in 1836, by Frémy, but an industrial application of his experiments was not attempted until 1840, when George Gwynne patented a process for decomposing fats with sulphuric acid. This process failed on account of the purification of the fatty acids obtained, since for the induction of saponification with sulphuric acid a further operation was required, namely, distillation of the fatty acids with superheated steam. The process of decomposing the fats with sulphuric acid and distilling the fatty acids by the use of superheated steam was patented, in 1842, by William Colly Jones and George Wilson.

Saponification with sulphuric acid may be effected by two different methods. According to one, the sulphuric acid is allowed to act for several hours upon the heated fat, while according to the other, the fat heated to between  $220^{\circ}$  and  $230^{\circ}$  F. is, for a few minutes only, brought in contact with a few per cent. of sulphuric acid of  $66^{\circ}$  B. and saponification thus started is finished by boiling for several hours with water containing sulphuric acid. The latter process is preferable; when carefully conducted good products are obtained and there is less loss than in the former method.

Saponification with sulphuric acid yields a larger quantity of candle material than the autoclave process—hence more

solid fatty acids and consequently less oleic acid—which proves that a portion of the latter is converted into solid material.

Connstein, Hoyer, and Wartenberg have recently made experiments with the object of utilizing for industrial application the well-known fact that certain ferments are capable of decomposing fats. They used for this purpose the ferment contained in the castor bean. According to Connstein, the process is as follows:

Fresh castor beans are ground in a mill or between stone rolls, and then intimately mixed with the melted fat to be decomposed. The hulls may eventually be removed by decanting or sifting, though it is not necessary. For every 220 lbs. of fat 22 lbs. of ground beans are required. To the mixture of fat and beans, 40 to 100 per cent. of acidulated water is added. For acidulation acetic acid (7 to 21 ozs. of glacial acetic acid in 220 lbs. of water) is preferably used. Fat, beans and acidulated water are then thoroughly mixed by stirring and allowed to stand at the ordinary temperature of a room. The ferment soon becomes active and in about 2 hours 20 to 40 per cent. of the fat is split. By carefully stirring the mixture, for about 5 minutes every hour, by means of a crutch or mechanical stirrer, or by blowing in air, a splitting of at least 85 to 90 per cent. is, in 24 hours, secured. This will be satisfactory to most soap boilers, and it will but seldom be attempted to obtain an increased splitting from 90 to 95 per cent. by allowing the mixture to stand for a longer time.

When splitting is completed the next operation, namely, the separation of the mixture into glycerin water, fatty acids and particles of beans begins. For this purpose the use of heat and dilute sulphuric acid is recommended. The mixture is heated by direct or indirect steam, eventually also by a direct fire, to 176° F., and dilute sulphuric acid—for every 220 lbs. of fat about 4.4 lbs. 25 per cent. sulphuric acid—introduced. It will then be noticed that the particles of beans floating in the mass fall to the bottom as if they were coagulated, so that in a

short time three sharply separated layers will be observed ; 1, on the bottom of the vessel a layer of acid glycerin water ; 2, floating above it a layer of emulsion consisting of particles of beans, glycerin water and fatty acid, and, 3, above this, clear fatty acid. The proportional quantities are about as follows : If for instance, 220 lbs. of fat, 142 lbs. of acidulated water and 15.4 lbs. of fresh beans (after removing by sifting 6.6 lbs. of hulls) have been used, the quantity of 1—the acid glycerin water on the bottom of the vessel will amount to about 110 lbs.; that of 2—the layer of emulsion—to about 48.4 lbs.; and that of 3—clear fatty acid—to about 209 lbs. A yield of about 138.6 lbs. of glycerin water and 220 lbs. of fatty acid (taking into consideration the oleic acid derived from the castor beans) might have been expected.

Thus, of the expected yield, there can at once be obtained in an available form, about 80 per cent. of glycerin water and 95 per cent. of fatty acid. The missing 20 per cent. of glycerin water and 5 per cent. of fatty acid are contained in the layer of emulsion. The latter is brought into a special vessel, mixed with warm water, and stirred. The water dissolves the glycerin in the layer and is then drawn off from the bottom. The 5 per cent. of fatty acid is best obtained from the washed middle layer by saponifying the entire layer in the ordinary manner with soda lye and separating the soap with common salt. According to Connstein's statement the particles of beans and other impurities remain in the spent lye. This, however, is not correct, since in salting out a considerable portion of the hulls of the seeds remains in the soap. This drawback is now overcome by the use of a castor bean extract in place of comminuted castor beans.

Twitchell obtains a nearly complete saponification of the fats and oils by the addition of 1 to 2.5 per cent. of a sulpho-aromatic combination. The latter is obtained by the action of concentrated sulphuric acid upon a solution of oleic acid in an aromatic hydrocarbon. With the use of benzol a product is obtained which Twitchell considers as corresponding to

$C_6H_4(SO_3H)(C_{18}H_{35}O_2^3)$ . Saponification takes place by heating in a current of steam, but still more readily so in the presence of a few per cent. of free fatty acids as a starting material, they being especially suitable to induce saponification.

The chemical process which takes place in this method has not yet been completely elucidated. Lewkowitch\* explains the action of this reagent by its ability to emulsify the glycerides. He is of the opinion that during the treatment with steam sulphuric acid is formed so to say *in situ nascendi*, which then acts upon the glycerides, sulpho-combinations being at the same time formed, and the latter are then more readily saponified with water than the glycerides themselves. This method differs, however, essentially from the process of saponification with sulphuric acid in that no oleic acid is converted into solid material. For this reason the fatty acids obtained by this process have the same composition as those prepared by the autoclave method; however, they readily acquire a dark color when not excluded from the access of air.

The products of saponification are, according to the process employed, glycerin and fatty acids, or glycerin and soap. The fatty acids, as far as they are of importance to the soap manufacturer, will be referred to later on. A few words may here be said in regard to glycerin and the general properties of soap.

*Glycerin.* In the manufacture of stearin, glycerin is obtained in the form of a watery solution showing at the utmost 4° to 5° B. In the ordinary lime saponification the solution is so thin as to scarcely act upon the hydrometer. It contains more or less impurities according to the method by which it is obtained, sulphuric acid being the principal impurity in the product resulting from acid saponification. The sulphuric acid is fixed by the addition of slaked lime until alkaline reaction takes place, and the glycerin is then evaporated with indirect steam. During evaporation the greater part of the

\* Chemische Technologie und Analyse der Oele, u. s. w. 1905, S. 642

sulphate of lime separates, while a heavy scum containing much of the impure matter forms on the surface and is removed with perforated ladles. When the glycerin has been evaporated to the desired concentration, generally 25° to 28° B., the steam is shut off and after allowing the sulphate of lime to settle, the supernatant clear glycerin is drawn off by means of a siphon. The sediment is washed with water and the latter evaporated together with a fresh lot.

The evaporation of glycerin in open vessels, however, involves loss, and some manufacturers evaporate in a vacuum, an apparatus with revolving cylinder, constructed by Leon Droux, being very suitable for the purpose. It will be described later on in speaking of the utilization of spent lyes from grained soaps.

The glycerin liquor obtained by the ordinary process of saponification by lime, or in the autoclave, is turbid from finely divided particles of lime, which cannot be removed by filtering. Hence the glycerin-liquor is diluted with dilute sulphuric acid until it shows a slightly acid reaction; the lime-soap is decomposed and the fatty acids separate on top. The glycerin solution is then filtered, neutralized with calcium carbonate, and finally evaporated to the desired concentration.

Crude glycerin of commerce is of a more or less dark-brown color and contains more or less impurities consisting of volatile fatty acids, hydrocarbons, and inorganic salts, the latter being derived from the lime and water used in saponification. Crude glycerin varies considerably according to whether it results from acid or autoclave saponification, the former having usually a peculiar, disagreeable tang, and shows strong dichroism. These properties are no doubt due to dissolved hydrocarbons. Glycerin produced by autoclave saponification has generally a purer taste and can be more readily decolorized by animal charcoal. However, the above-mentioned characteristics cannot be considered as absolutely sure, as in some cases it is impossible to say with any degree of certainty by which process of saponification the crude glycerin has been

obtained. By tribasic lead acetate (Goulard's extract) all the impurities naturally occurring in crude glycerin are precipitated, but not any additions, such as sugar, which may have been made. Now, since crude glycerin obtained by acid saponification contains on an average more organic impurities than the product resulting from autoclave-saponification, it may generally be assumed that a crude product which yields a heavy precipitate with tribasic lead acetate is derived from acid saponification.

While crude glycerin can be used for many technical purposes, for others it has to be purified. This is effected either by filtration through animal charcoal or by distillation with superheated steam. The product obtained by the first method is generally termed in commerce "refined glycerin." It is however, not perfectly pure as it always contains a larger or smaller quantity of lime salts and traces of butyric acid, the latter being recognized by the odor on rubbing a sample upon the hand. Chemically pure glycerin can only be obtained by distillation or by crystallization, the latter process being, however, too slow to be remunerative.

Chemically pure glycerin should not become turbid by the addition of ammonia and ammonium oxalate, nor of nitric acid or nitrate of silver. It should not smell when rubbed upon the hand nor become colored by the addition of nitrate of silver without nitric acid. The so-called "simple distilled glycerin" generally shows slight turbidity on adding ammonia and ammonium oxalate, as well as nitric acid and nitrate of silver. These slight traces of foreign salts, however, do not injure the value of the glycerin for technical or other purposes.

The numerous applications which glycerin has found are chiefly based upon the following important properties it possesses: It is non-fermentable, possesses great solvent power for a number of substances, is non-drying, and its watery solutions, even when very weak, freeze only at low temperatures. On account of its softening and smoothing effect on the skin, it is used in the preparation of toilet soaps and cosmetics.

By treating glycerin with a mixture of concentrated nitric and sulphuric acids, nitro-glycerin—a very explosive body—is formed; it is the basis of dynamite and similar blasting agents. The glycerin which is to be used for the preparation of nitro-glycerin should not contain any chlorine or lime, and must be as free as possible from organic impurities.

In the manufacture of soap the glycerin passes either into the soap, as in half-grained and soft soaps, or into the spent lye, as in grained soaps. Its recovery from the spent lye is connected with difficulties on account of the presence of large quantities of common salt. The methods used for the purpose will be given later on.

*Soaps.*—The ordinary soaps of commerce are, generally speaking, mixtures of the potassium and sodium salts of stearic, palinitic, and oleic acids, and if cocoa-nut and palm-oils are used as an addition, also of lauric acid. They are soluble in ether, benzol, and petroleum ether. Their alcoholic solutions are transparent and admit of filtering; at a certain degree of concentration they form a jelly. By evaporating them to dryness the soap is left behind as a clear, transparent, solid mass, free from crystalline admixtures.

Soaps show a peculiar behavior towards water. While they dissolve to a clear solution in boiling water, a solution in cold water is peculiarly turbid and opalescent. If, according to Knapp, a piece of soap is placed in a wire net or cloth, and suspended just below the level of cold water in a tall vessel, a certain amount of the constituents of the soap passes gradually into solution, while the residue remains as a coherent piece of the original shape. This insoluble residue remains even when the water is repeatedly renewed. It presents a wood-like fibrous appearance, and forms, so to say, the skeleton of the original soap-mass. A piece of soap placed in cold water and frequently shaken or stirred with it, dissolves to a whitish, milky, opaque mass in which floats a residue which renders the mass turbid and, when moved in the light, exhibits a silky luster which is frequently quite remarkable.

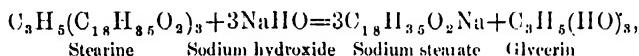
The fluid shows great tendency towards forming an abundance of foam which remains standing for a long time. Solution in cold water is, therefore, incomplete. When hot clear aqueous soap solution is diluted with water, it becomes turbid and, on shaking, a foam which holds for a long time is formed. This turbidity is caused by the dissociation of the neutral salt. This dissociation—*the hydrolysis of the soap*—takes place very gradually and depends on the quantity of water added as well as on the temperature. An addition of free alkali retards dissociation, as well as an addition of alcohol. Absolute alcohol—even 90 to 95 per cent. alcohol—dissolves neutral soap without dissociating it. Chevreul found that a solution of neutral potassium stearate,  $C_{18}H_{35}O_2K$ , in 20 parts of boiling water, yields, after adding 1000 parts more of boiling water and allowing to cool, an acid potassium stearate of the composition  $C_{18}H_{35}O_2K.C_{18}H_{36}O_2$ , while potassium hydrate and an almost imperceptible trace of stearic acid remain in solution. Alkali is also withdrawn from the potassium stearate by a larger quantity of cold water, and acid salt remains behind. Neutral sodium stearate dissolved in 2000 to 3000 parts of hot water and, when allowed to cool, yields the acid salt  $C_{18}H_{35}O_2Na.C_{18}H_{36}O_2$ , which is insoluble in water. There also exist, according to Chevreul, acid oleates, and he obtained from weighed quantities of oleic acid and potassium hydrate, potassium biolate in the form of a substance soluble with great difficulty in water and scarcely capable of being filtered. On the other hand, regarding the action of water upon neutral oleates he says: "The potassium oleate is deliquescent and for the spontaneous decomposition of its solution it has to be largely diluted with water and exposed for quite a long time to a low temperature."

Hence, according to Chevreul, the effect of water upon soap is simply a splitting-off of alkali. This view is, however, contested by many chemists, especially by Rotondi,\* A. Fricke,†

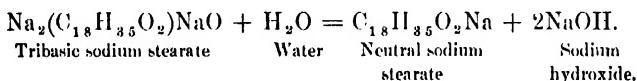
\* Seifenfabrikant, 1886, S. 284.

† Dingler. Polytech. Jour. 209; S. 46.

and M. Dechan and T. Maben.\* Rotondi is of the opinion that soaps are not decomposed into alkaline oleate and free alkali, but into alkaline oleate and a basic alkaline oleate. Fricke considers it probable that in grained tallow soap the sodium oleate dissolves, while the sodium palmitate or stearate separates in nacreous threads. He further found that the residue left by lixiviating soap in cold water is even insoluble in hot water, and that alcohol dissolves only 0.48 per cent. of it, it being, however, freely soluble in the hot solution of the extracted portion. Dechan and Maben are of the opinion that by the saponification of fats not only neutral salts are formed, so that the process is not only according to the following equation:



but also basic salts, hence, salts of the formula  $\text{Na}_2(\text{C}_{18}\text{H}_{35}\text{O}_2)_n\text{NaO}$ . By treating such a salt with cold water, decomposition takes place according to the following equation :



Rotondi has thoroughly investigated the action of water upon soap. He sought to solve the question with the assistance of dialysis to which he subjected solutions of pure Marseilles soap, and arrived at the following conclusions: 1. Neutral soaps with an alkali as a basis ( $C_nH_{2n-1}MO_2$ ) are decomposed by water into basic soaps ( $C_nH_{2n-1}MO_2OMH$ ), soluble in hot and cold water, and in acid soaps ( $C_nH_{2n-1}MO_2C_nH_{2n}O_2$ ) insoluble in cold, and very sparingly soluble in, hot water. 2. The decomposition of neutral soaps takes place more freely in hot than in cold water, and more rapidly or more slowly according to the concentration of the solution and the prevailing temperature. 3. The basic soaps diffuse freely,

\* Seifenfabrikant, 1886, S. 91.

but the acid ones with difficulty. 4. The solution of basic soap obtained by dialysis may contain some neutral soap, which can be decomposed into basic and acid soaps, until the solution finally contains only basic soap. 5. By the decomposition of neutral soaps by water neither alkaline hydroxide nor alkaline carbonate is liberated, which is readily shown by precipitating the neutral soaps with common salt and analyzing the fluid after filtering.

F. Kraft and A. Stern \* subjected neutral sodium palmitate to the action of varying quantities of water. In the different experiments 2 grammes of finely triturated common salt were boiled with 200, 300, 400, and 900 times the quantity of pure water, whereby heavy foaming always took place, and the fluid represented a turbid solution, apparently holding in suspension extremely fine drops of melted free fatty acid comparable with milk much diluted with water. On cooling, a micro-crystalline mass with nacreous luster always separated. This mass, according to the quantity of water used, could be more or less readily filtered off, the process sometimes requiring one or two days, and succeeding most rapidly, without clogging the filter and without turbid filtering, by allowing the precipitate to stand for some time, shaking occasionally. In this manner the above-mentioned chemists found that the neutral sodium palmitate when boiled with 900 times the quantity of water, forms, on cooling, the acid sodium palmitate ( $C_{12}H_{22}O_2Na.C_{16}H_{32}O_2$ ), while, on the other hand, with the use of smaller quantities of water mixtures of acid and neutral soaps are obtained. They further found that in the alkaline filtrates obtained from the separation of the acid soaps, a noticeable precipitation or turbidity was never produced by the addition of mineral acids, such as would have been the case if the alkaline oleates had been decomposed into non-soluble acid, and readily soluble basic, salts. The neutral sodium stearate behaves in the same manner as sodium pal-

\* Seifenfabrikant, 1894, S. 669.

mitate. Pure neutral sodium oleate ( $C_{18}H_{38}O_2Na$ ), according to Krafft and Stern, dissolves clear in about 10 times its quantity of cold water, while heat is required for the stearate or palmitate. While, however, the solution of the sodium stearate or palmitate is rendered turbid by much hot water and is perceptibly decomposed, the solution of the sodium oleate, when more water is added, remains at first clear, and a very slight turbidity consisting very likely of fine drops of oleate is only produced on the addition of 200 times the weight of water; this turbidity is not very marked even with 900 parts of water, and disappears immediately on the addition of a small quantity of alkali. However, the acid sodium oleate ( $C_{18}H_{38}O_2Na \cdot C_{18}H_{34}O_2$ ) behaves in an entirely different manner. It is a solid salt and dissolves immediately when brought into a large quantity of water, a heavy milky turbidity consisting of innumerable extremely fine drops of oleate, clearly distinguishable under the microscope, being formed. Hence, while from what has been above said, alkaline stearate and palmitate are decomposed only by hot water, with acid sodium oleate this is the case at the ordinary temperature.

The alkaline salts of the lower homologues of the saturated fatty acids behave in a different manner from the corresponding salts of stearic and palmitic acids, hydrolytic splitting decreasing as the molecules become smaller. While Krafft found that 1 gramme sodium stearate in 300 parts of water splits off soda lye equivalent to 0.0171 Na, and 1 gramme sodium palmitate, under the same conditions, soda lye equivalent to 0.0143 Na, he says in regard to lauric acid: "The separation was less abundant than with the two higher acids." To the investigations of C. Stiepel \* we are indebted for the knowledge of the interesting fact that there are soap-forming fatty acids—caprylic and pelargonic acids—whose sodium salts in water suffer no alkaline splitting, while the latter is very slight with soaps of caproic acid.

\* Seifenfabrikant, 1901, S. 1186.

Stiepel also investigated the behavior of a number of natural fats in aqueous solution and found that, as regards their behavior, the fatty acids of natural fats which consist of palmitic, stearic and oleic acids, conform in every respect to the corresponding behavior of the individual fatty acids, *i. e.*, the soaps of these fats are in a large quantity of water decomposed so that about one-half of the alkali present is split off. The other high molecular fatty acids of the natural fats—linoleic acid, physetoleic acid, etc.—behave in a similar manner, but in the case of palm-kernel oil soap, and still more so, in that of cocoanut oil soap, the presence of less, or not at all dissociated, soaps of lower fatty acids makes itself felt. While in the case of tallow soaps which contained 7.64 per cent. Na, 4.03 per cent. soda was split off as caustic soda, hence, 52.5 per cent., in that of palm-kernal oil soaps which contained 8.74 per cent. Na only 3.67 per cent. Na, *i. e.* 29.6 per cent. was split off, and in that of cocoanut oil soaps, which contained 10.1 per cent. Na, only 33 per cent. Notwithstanding the higher total content of alkali, 100 grammes of cocoanut oil soap split off less alkali (3.3 grammes) than tallow soap (4.05 grammes).

If by evaporation water be withdrawn from soap solutions they become, as concentration increases, more thickly-fluid and viscid, and finally the mass can be drawn into threads. When the solutions in a thickly-fluid state are allowed to cool, a jelly or a completely solid mass is, according to circumstances, formed. The jelly, and even the soap solution congealed to a hard solid mass, contain an abundance of water, a considerable portion of which is mechanically fixed, and another chemically, and is retained at 212° F. This affinity for water varies very much with soaps from different fats, and involves distinctions of great practical importance. It shows itself, on the one hand, in the quantity of water which the soap in congealing is capable of fixing—it being accepted as a rule that soaps from solid fats can fix more water—and, on the other, in the behavior towards the mois-

ture of the air. Dry potash soaps absorb water with avidity from the air, while moist soda soaps dry out in the air. The nature of the fatty acids exerts a similar influence, oleic acid soaps being far more hygroscopic than solid fatty acid soaps.

When exposed for a longer time to the air,

One hundred parts by weight of sodium stearate withdrew 7½ parts by weight of moisture.

One hundred parts by weight of potassium stearate withdrew 20 parts.

One hundred parts by weight of potassium oleate withdrew 162 parts.

The soaps of oleic acid with potash as a basis, even when at first solid, are gradually changed to a jelly on exposure to the air.

The soap solutions when congealed to a hard, solid mass do not present, when their content of water does not exceed a certain degree, a homogeneous mass, there appearing on the contrary crystalline veins in an opaque, amorphous ground. The soap maker calls this "grain and flux." The crystalline veins are without doubt formed by acid alkaline stearate and acid alkaline palmitate.

In the presence of other bodies having great affinity for water, the behavior of soaps towards water undergoes considerable modifications. A piece of soda soap placed in a cold concentrated common salt solution floats in it like mercury; it does not dissolve in it but remains perfectly solid. On heating the salt solution the soap softens to a thick viscid mass which floats, sharply separated from the salt solution, on top and, on shaking, is divided into flakes which again collect when at rest. The soap thereby yields water to the common salt solution, but only to a certain amount, which it retains fixed. Hence soda soap is insoluble in cold and hot concentrated common salt solutions.

Soap solution and common salt solution mix only when diluted to a certain degree. For most soaps the solution has to be much diluted, less dilution being required only for soaps

from palm-kernel and cocoanut oils. Solutions of greater concentration separate in layers one above the other. When common salt is added to a solution of soap in water it withdraws from the latter a certain quantity of water for its own solution, and separated solutions of salt and soap are formed.

Similar effects, though to a less degree, are produced by solutions of potassium acetate, ammonium chloride, potassium chloride, sodium carbonate and sodium sulphate. Most all kinds of soap are soluble in weak caustic lyes, but not in more concentrated ones.

Potash soaps are decomposed by sodium salts, for instance, common salt, Glauber's salt, etc., and the corresponding potassium salt—potassium chloride, potassium sulphate—and soda soap are formed. This was formerly the only means of preparing hard soaps. The fat was saponified with potash lye and common salt was added to exchange the bases. This exchange of bases, however, is not complete as the soap thus prepared always contains potash and is in consequence somewhat softer and more soluble than that boiled with pure soda lye.

The processes which take place in the conversion of soft potash soaps into hard soda soaps have been thoroughly investigated by C. R. Alder Wright and C. Thompson. They also found that the reciprocal transposition between sodium chloride and potassium oleate is only a partial one, but proceeds in such a manner that the partition of the two acids (fatty acid and hydrochloric acid) takes place in accordance with their numbers.

In making their experiments, Wright and Thompson treated the fatty acid to be examined at the same time with the quantity of potash and soda required for neutralization, hence, 1 equivalent of acid to 2 equivalents of alkali. A weighed quantity of fatty acid was brought into the required quantity of mixed lyes, then heated on a water bath and, when melted, thoroughly shaken. The fluids were allowed to clarify by standing and a portion of the clear solutions was used for

analysis. The results obtained with the different fatty acids used for the experiments were as follows:

Fatty acids used.	Per cent. of the fatty acids converted into	
	Soda soap.	Potash soap.
Stearic acid . . . . .	51.2	48.8
Oleic acid . . . . .	50.8	49.2
Crude stearic and oleic acids (tallow) . . . . .	51.5	48.5
Crude stearic, palmitic and oleic acids (palm oil and tallow) . . . . .	48.2	51.8
Crude lauric acid (coconut oil) . . . . .	49.7	50.3

From this it may be inferred that in treating a potash soap with the equivalent quantity of soda nearly as much soda soap is formed as in treating soda soap with potash, while about one-half of the alkalies used remain unchanged. The result of direct experiments showed that in treating potash soaps with the equivalent quantity of sodium hydroxide, 48.8 per cent. of them is converted into soda soap, and in treating soda soap with the equivalent quantity of potassium hydrate, 46 per cent. of it is converted into potash soap.

Hence, while in treating a soap with another free alkali a partition of the bases in the fatty acids takes place in the proportion of 1:1; the proportion is a different one when an alkaline carbonate is allowed to act upon the fatty acid combination of another alkali. The determination of the products of reaction in this case was effected by shaking the soap prepared by heating lye with fatty acid with the equivalent quantity of solution of the other alkaline carbonate. It was then evaporated on the water-bath and the soap withdrawn from the residue by strong alcohol. Hence, it follows, as will be seen from the following table, that the proportion of potash contained as potash soap in the final product to sodium (as soda soap) is far greater than that of the potash as potassium car-

bonate to soda in the form of sodium carbonate, no matter whether potash soap was treated with sodium carbonate or soda soap with potassium carbonate.

Fatty acids used.	Soda soap treated with $K_2CO_3$ . Per cent. of total fatty acids present equivalent to the added		Potash soap treated with $Na_2CO_3$ . Per cent. of total fatty acids present equivalent to the added	
	$K_2CO_3$ .	$Na_2CO_3$ .	$K_2CO_3$ .	$Na_2CO_3$ .
Stearic and oleic acids . . .	10.1 45.7 100.00	8.0 34.4 97.95	— — 100.0	— — 4.3
Stearic, palmitic and oleic acids (palm oil and tallow) . . .	104.2 57.2 108.0 52.8	99.00 52.1 90.8 46.4	1000.0 — 177.0 —	15.0 — 9.5 —
Crude lauric acid (coconut oil) {	114.8	87.9	197.0	6.2
Crude ricinoleic acid (castor oil) {	50.00 100.00	48.4 93.8	— 205.0	8.2

This explains the effect produced as regards texture and grain by the treatment of soda soaps with potash solution; the hard soda soaps being partially converted into soft potash soaps. Now, while in the presence of fatty acid and carbonic acid, on the one hand, and potassium and sodium on the other, the reaction between the components takes place so that potash soap (and sodium carbonate) are preponderatingly formed, the proportion is just the reverse when alkaline chlorides are used in place of the carbonate.

In making experiments relative to this matter, a known quantity of fatty acid was divided into two equal portions, and one of them neutralized with potash lye and the other with soda lye. Both the soaps were then mixed and dissolved in hot water (150 molecules to 1 molecule of the soap mixture). The solution was then treated at 212° F. with a mixture of equivalent quantities of potassium and sodium chloride (20 molecules of the mixture) by introducing the

mixture in the form of powder, with constant shaking, into the solution. The results were as follows:

Fatty acids used.	Per cent. fatty acids contained as		Molecular proportion of the soda soap to the potash soap.
	Potash soap.	Soda soap.	
Pure oleic acid . . .	38.0	62.0	1.63 : 1
Crude ricinoleic acid . . . .	17.8	82.2	4.6 : 1
Stearic, oleic and sylvic acids .	17.2	82.8	4.8 : 1
Crude lauric acid . . . .	15.1	85.9	5.7 : 1

Further experiments were made regarding (a) the quantities of potash soaps which are separated from a solution in M molecules water by N molecules sodium chloride to 1 molecule potash soap, and (b), the quantities of soda soap from a solution of M molecules water by N molecules potassium chloride to 1 molecule soda soap. The results of the experiments were as follows:

Fatty acids.	M	N	(a). Potash soap separated with NaCl.	(b). Soda soap separated with KCl	
				Potash soap.	Soda soap.
Stearic and oleic acids . {	100	5	10.5	89.5	79.1
	200	20	5.1	94.9	82.1
Stearic palmitic and oleic acids (palm oil and tallow)	200	20	3.8	96.2	95.8
Crude lauric acid . . . .	200	20	5.4	94.6	74.8
					25.2

The soaps of the alkalies are completely decomposed by the salts of the metals of the earths and of the heavy metals, because the earthy oleates, etc., which are formed are insoluble in water.

In aqueous solutions of ammonia soaps, dissociation takes place to a far greater extent than is the case with the salts of the alkali metals. The aqueous solutions lose ammonia by heating and for this reason neutral ammonia soaps can scarcely be prepared on a large scale. By allowing ammonia soap to stand under a glass bell over sulphuric acid, ammonia is at first rapidly given up until the remaining quantity contains about one-half the amount of ammonia of a neutral soap. By allowing the acid soap thus formed to stand for a longer time it further loses ammonia, but much less rapidly than at first. The acid soaps of stearic and lauric acids appear in this respect to keep less constant than those of oleic and ricinoleic acids. By boiling ammonia soap solution, complete decomposition takes place so that the fatty acid as such finally floats upon the water.

It cannot be very readily decided on what the detergent action of soap depends. Chevreul showed that for the saponification of the fats and oils a certain quantity of alkali is required. With the use of a smaller quantity of alkali the fat not attacked forms, as he observed, if not a chemical combination, at least an intimate mixture with the soap, and this mixture yields with water an emulsion which no longer spots fabrics. An emulsion of this character is formed in degreasing whereby the fatty bodies which are removed from the stuffs are not saponified. Hence, according to Chevreul, the detergent action of soaps depends essentially on their ability to emulsify fats. According to Berzelius, the use of soap for washing depends 1, on its power as an emulsion-like solution to take up from the stuff fatty matter, which thereby dissolves in the soap water, and 2, upon the facility with which its dissolved salts yield their alkali. Neutral oleate is at the ordinary temperature more readily decomposed than carbonic acid

is expelled from alkaline carbonates, and is favorably distinguished from the caustic alkalies, which would be cheaper washing agents, by saving the clothes, skin, etc. Other chemists explain the action of soap as a purely chemical one. Kobbe says: "The detergent action of soap is due to the fact that the alkaline oleates when in contact with a large quantity of water are decomposed to free alkali and an acid salt which is insoluble in water and forms with it a strong foam. The alkali carries away the fatty dirt of the objects treated with soap, and the foam contributes to its mechanical removal."

According to Rotondi, the detergent action of soaps depends on the fact that they are decomposed by water into basic soaps which are soluble in cold, and still more so, in hot water, and into acid soaps, and that the basic soaps possess the property to emulsify fats. This theory, however, is not tenable as Kraft and Stern have proved the non-existence of basic salts.

Knapp's theory deserves attention. He is of the opinion that the detergent action of soap is principally due to the great power of wetting substances possessed by soap solutions, which in this respect surpass nearly all other liquids. The dissolving effect of soap upon grease and dirt presupposes an extremely intimate contact between the substance to be dissolved and the solvent, and this contact is rendered possible by the wetting power of soap solutions. Soap-water penetrates tissues, etc., with greater ease and more completely and wets the surfaces more thoroughly than mere water; it readily dislodges the condensed layer of air upon the surface, and by capillarity forces itself between the surface of the substances to be cleansed and the adhering particles of dirt, dissolving and removing them.

A very original explanation of the detergent powers of soap has been advanced by Professor Jevons, of London. He observed that inorganic as well as organic particles suspended in water remain in a peculiar shaking and trembling motion,

provided their diameter does not exceed  $\frac{1}{500}$  inch. To this phenomenon he applied the term "pedesis," a Greek word meaning a motion by leaps or bounds. Upon this fact Jevons bases his explanation of the effect of soap. We would, therefore, have to suppose that the particles of soap suspended in the water dash at the particles of dirt, thus dissolving and washing them away. Nearly all substances soluble in water have, it is claimed, the power of interrupting pedesis, which, according to Jevons, explains the fact that distilled water or pure rain-water possesses great cleansing power, since it produces a high degree of pedesis, while the comparatively small effect of hard water is due to a considerable decrease of pedesis, in consequence of the earthy salts dissolved in the water.

That the action of soap is not a purely chemical one is proved by the fact that soap from sylvic acids and lower members of the fatty acid series dissociate not at all, or only very slightly, in aqueous solution, but nevertheless possess detergent powers. Hence, the opinion may be expressed that the detergent action of soap rests in the first place upon a physical basis, though in most cases the free alkali already present in the soap or formed by splitting may also participate in the detergent action.

Soap possesses in a high degree disinfecting and deodorizing properties. According to experiments made by the Berlin Health Commission a solution of 1 part potash soap in 10,000 parts water completely prevents the development of anthrax bacilli. Whether this disinfecting property depends only on the free alkali in the soap or on that splitting-off by solution, has not yet been determined.

## CHAPTER III.

### OCCURRENCE AND MANNER OF OBTAINING AND PURIFYING FATS AND FAT OILS.

*Occurrence and manner of gaining fats and oils.*—The fats are widely distributed throughout the animal and vegetable kingdoms. In the animal organism fat occurs in all tissues and organs and in all fluids except normal urine. In plants it is found partly scattered throughout the entire plant and partly heaped up in certain organs, especially in the seeds and cotyledons.

The vegetable fats occurring in moderate climates are liquid at the ordinary temperature. They are obtained by grinding or crushing the seed, packing the meal thus produced in cloth bags and subjecting the latter to strong pressure, or by extraction with a solvent such as carbon disulphide, benzine, and, in recent times, carbon tetrachloride.

Most of the vegetable fats occurring in warm climates are solid at our ordinary temperatures. They are mostly obtained in the country of their origin by boiling the oleaginous seeds and fruits with water. Some of them, such as palm-kernels, copra (the dried pulp of the cocoonut), etc., are also exported to Europe and America, where the fat is obtained by pressure or extraction.

The fat from animal substances is generally obtained by heating over an open fire or with steam. With the use of the latter its action is mostly assisted by the addition of acids or caustic alkalies. In some cases the separation of the fat from the enclosing membranes is effected by steam under pressure in closed tanks. From bones the fat is frequently extracted with benzine.

*Purifying and refining fats and oils.*—The fats and oils, no matter by what method they are obtained, are never entirely pure; they always contain more or less foreign substances derived from the raw materials, which form an obstacle in their use for many purposes. The animal fats contain generally particles of flesh and blood, which, if not removed, would in a certain sense act as ferments and promote rancidity. The fats are, therefore, purified by remelting upon water over an open fire or with the assistance of steam.

Vegetable oils contain more or less mucilage and other substances in solution, which give to the oil a milky turbidity. These foreign substances exert a disturbing influence if the oil is to be used for lubricating or illuminating purposes, it becoming soon rancid, and in burning chokes up the wick and deposits soot. It is, therefore, subjected to a purifying process called "refining," and the oil thus treated is termed "refined oil."

Oils for illuminating purposes are best refined by means of concentrated sulphuric acid, the latter possessing the property of carbonizing most organic substances with which it is brought in contact. It withdraws hydrogen and oxygen from these organic bodies, a carbonized black mass remaining behind which is insoluble in the ordinary solvents. In this manner the foreign substances contained in the oil are also destroyed. To be sure, the oils themselves do not resist the energetic action of concentrated acids. However, by using only a small quantity of concentrated sulphuric acid the latter acts especially upon the admixed foreign substances, carbonizing them. According to the condition of the oil the quantity of sulphuric acid required varies from 1 to 1½ per cent., if the oil is treated at the ordinary temperature. By heating the oil by steam to between 122° and 140° F. less than 1 per cent. acid will in most cases be required. However, the oil must not be kept too cold during the entire operation, as it then becomes too thickly-fluid and the substances carbonized by the sulphuric acid settle with difficulty.

The sulphuric acid is added in a thin stream and whilst constantly stirring. The oil assumes a dark green color. Stirring is continued until a drop of oil placed upon a porcelain plate shows that the carbonized substances have coagulated to a black flake floating in the clear yellow oil. Stirring is then discontinued and the oil allowed to stand quietly six to twelve hours. When the carbonized substances have settled on the bottom the oil is drawn off into a large vat, and one-fourth to one-third of its volume of hot water added. The whole is then moderately stirred for a quarter of an hour, and allowed to stand until the oil separates from the water. The latter is then drawn off through a cock placed immediately above the bottom of the vat, and the washing of the oil repeated once or twice to remove the last traces of sulphuric acid adhering to it. For the complete attainment of this object, it has been recommended to add some lime-paste or chalk to the water for the neutralization of the sulphuric acid. Such addition is, however, not necessary, and is rather a disadvantage, since the lime is apt to saponify some of the oil, and chalk forms a disagreeable froth in consequence of the development of carbonic acid. A small quantity of soda may, however, be added to the last wash-water.

After the separation and drawing off of the oil from the last wash-water, it still contains some aqueous particles in suspension. They will settle, and the oil be rendered entirely clear by storing for some time at not too low a temperature. Clarification is effected still more quickly by adding some common salt to the oil before storing it.

Treatment with sulphuric acid is, no doubt, the best method for refining oils for illuminating purposes; for oils for lubricating purposes it is, however, not suitable, or at least oils thus refined must be subjected to special treatment. While the opinion, so frequently expressed, that oils treated with sulphuric acid always retain some of it, is not correct, because the sulphuric acid can be readily removed by boiling with water, oils thus refined always contain free fatty acids, and

must, therefore, be rejected as lubricants, if not previously subjected to a special process to free them from acid. This is effected by various methods. One frequently employed consists in treating the oil with calcium carbonate (marble powder). This method is, however, not suitable, as the carbonate of lime will only remove traces of sulphuric acid, but not the free fatty acids. By another process the oil is treated with a few per cent. of lye. This, however, has the disadvantage of the soap-paste formed settling with difficulty. It is, therefore, best to use concentrated solution of soda and boil the whole with steam. Sodium aluminate is now also frequently used.

To avoid the formation of free fatty acids, Bareswil has proposed to treat the oils with alkalies instead of acids. The oil is compounded with 2 to 3 per cent. of concentrated potash or soda lye, then thoroughly stirred, and gradually heated. If, however, pure caustic lye is used, we have again to contend with the drawback of the oil clarifying with difficulty. It is therefore better to use lyes not entirely caustic, and even in this case clarification is frequently connected with difficulties, and entirely clear oil is generally only obtained by filtration, which is usually effected through layers of gravel, the upper one consisting of coarse gravel, the next one of finer, and the last one of sand.

*Bleaching of oils and fats.*—The methods used for bleaching oils and fats are generally the same as in other industries: Air and light, and chemical agents; among the latter being lye, sulphuric acid, sulphurous acid, chlorine, potassium permanganate, and potassium dichromate, in connection with acid.

Light has a bleaching effect upon all organic coloring matters, this action being based partly on an oxidation and partly on a disoxidation of the coloring matter. Of great importance is the action of sunlight upon essential and fat oils, as they are not only bleached, but the first are also quickly resinified, and the latter soon become rancid. Small

quantities of oil are frequently bleached by exposure to air. For this purpose the oil in carboys or in flat zinc boxes is exposed to the sunlight. On a large scale bleaching with air and light is frequently employed for palm-oil, which will be referred to later on.

In soap factories lye is much used for bleaching linseed and cottonseed oils, 5 to 10 per cent. of potash lye of 28° to 30° B., according to the condition of the oil, being usually employed for the purpose. The oil is first heated, best by direct steam, and the lye than added with vigorous stirring. The soap-paste separated in allowing the mass to rest is best used for dark soft soap. Where the demand for the latter is a sufficiently large one, bleaching with lye is simple and cheap. But where the demand is chiefly for pale, transparent soft soaps, bleaching with sulphuric acid is preferred. For 2000 pounds of oil 60 to 80 pounds of sulphuric acid of 66° B., previously diluted with 20 pounds of water, are used. (In diluting sulphuric acid with water, the acid is poured into the water.) The diluted acid is then poured in a thin stream into the oil, stirring constantly. The oil soon acquires a dark green color, and stirring is continued for twenty minutes. 120 to 160 pounds of warm water are then poured through a rose over the oil and thoroughly mixed with it by stirring. The oil now assumes a pale green color, which indicates the success of the bleaching process. The oil is then covered and allowed to stand for a few days for clarification. It has then a milky-white appearance, due to finely divided particles of water, and can be immediately used for the manufacture of pale soft soaps.

For bleaching with sulphurous acid, it is best to use acid sodium sulphite. It exerts, even in aqueous solution, a vigorous bleaching effect, and a still more powerful one when the solution is compounded with some sulphuric acid; the latter should be diluted and very gradually added as otherwise a too violent evolution of sulphurous acid is induced. For bleaching 100 lbs. of oil 1 to 1½ lbs. of acid sodium sulphite are required.

For bleaching with calcium chloride pour about ten times its weight of water over it, let the mixture stand for some time, frequently stirring it, and then allow the sediment to settle. Add, whilst constantly stirring, enough of the supernatant liquor to the oil to be bleached (previously compounded with some sulphuric acid) until it appears to be sufficiently bleached. It is then washed with hot water and clarified by filtering. This method of bleaching cannot, however, be recommended on account of the chlorine developed attacking the oils and fats too strongly.

Potassium permanganate, in connection with sulphuric acid, was formerly much used for bleaching palm oil. A solution of the permanganate strongly acidulated with sulphuric acid was added, whilst constantly stirring, to the melted palm oil, and the stirring continued  $\frac{1}{2}$  to 1 hour. The oil was then allowed to rest until the next day. The bleached oil settled upon the brownish fluid; this was removed and the oil repeatedly washed with hot water and then allowed to settle; pyrolusite (manganic peroxide) was frequently substituted for potassium permanganate. To the heated oil compounded with dilute sulphuric acid was added in small quantities and with constant stirring finely pulverized pyrolusite until the mass at first black, assumed a light appearance. The process was then finished in the same manner as with the potassium permanganate.

Both of these processes have, however, been universally superseded by that of bleaching with potassium dichromate, which was first made public by Watt in 1836. While he used potassium dichromate and sulphuric acid, hydrochloric acid is now generally substituted for the latter. Watt recommended the process, not only for palm oil, but also for tallow and other fats. Its execution is similar to that with potassium permanganate and sulphuric acid, and will be referred to more fully under palm oil. Watt also published a process to regain the potassium chromate used. The green liquor formed beneath the fat is drawn off, diluted with water, and then

compounded with thick lime-paste until the sulphuric acid is saturated, *i. e.*, until the acid reaction disappears. The gypsum formed is then allowed to deposit, the clear green solution drawn off, and after washing it once more, carefully precipitated with milk of lime until the green color disappears. The precipitate is thoroughly washed, dried, and heated to a red heat upon an iron plate. The powder gradually becomes yellow, and can be used for fresh bleaching operations by compounding it with sulphuric acid so that the latter slightly preponderates. This process of regaining the bleaching agent will of course only pay where very large quantities are used.

Hydrogen peroxide being at present manufactured on a large scale is now also used for bleaching oil, and has been especially recommended for bleaching olive oil foots. Commercial aqueous solution of hydrogen peroxide is known as ten-volume peroxide of hydrogen, it yielding about ten volumes of active oxygen, and, according to weight, is equivalent to from 2.5 to 3 per cent. The hydrogen peroxide solution should be intimately mixed with the oil. It does not act very energetically, and much more slowly than the previously-mentioned bleaching agents, several days being required before the oil is sufficiently bleached, and during this time it has to be frequently stirred. Upon many oils it exerts no bleaching effect whatever; linseed oil, for instance, which was finely discolored by the sulphuric acid process, was not bleached at all by hydrogen peroxide.

Animal charcoal has also been recommended for bleaching oil. It is, however, not well adapted for the purpose, as it very rapidly loses its bleaching power in contact with oil.

## CHAPTER IV.

### EXAMINATION OF FATS AND FAT OILS.

In examining fats and fat oils problems of varying natures may present themselves for solution. It may be required to determine the chemical constitution of a fat, its identity, its percentage of solid and liquid glycerides, glycerin, and free fatty acids, and an intentional or unintentional contamination with other fats or oils, or with foreign admixtures. The solution of the first problem—the chemical constitution of a fat or oil—belongs to science, and therefore does not require our attention here; the other examinations are, however, frequently required in the fat industry, and we will, therefore, refer to various methods in use. They may be divided into three classes: 1. *Organoleptic methods*; 2. *Physical methods*; and 3. *Chemical methods*.

1. *Organoleptic methods*.—The organoleptic means, *i. e.* odor, taste and color, are generally employed in commerce as criterions of the quality of an oil. Their use requires, of course, great experience, and, besides, they are by no means reliable, since the color, taste, and odor of oils not only undergo alteration by age, but vary according to derivation; linseed-oil from Russian seed having, for instance, a different taste from that from Indian seed. The odor of an oil is generally tested by rubbing a few drops upon the palm of the hand. It has also been proposed to carefully heat a few drops of the oil to be examined in a small porcelain dish, and when sufficiently cool to rub some upon the palm of the hand and smell, carefully comparing the odor evolved with that arising from a known pure sample of the same kind and quantity of the oil similarly treated.

However, the physical and chemical methods are only of actual value for the examination of fats, and even they are, in many cases, misleading.

*Sampling.*—If a larger lot of fat or oil is to be tested, it is first of all necessary to obtain a fair average sample. With oils this is comparatively easy. In case stearin has separated it must, as uniformly as possible, be distributed throughout the oil by stirring, or by rolling the barrel. A fair average sample of solid fats is not so readily obtained. According to Lewkowitsch,\* the following method is generally employed in sea-ports and factories: Several cylinders of fat, each at least 8 inches long and  $\frac{1}{2}$  inch thick, are taken by means of a sampler from each barrel, and the weight of the corresponding barrel is noted on these samples. The separate samples are then mixed in quantities corresponding to the net weight of the barrels, and the mass thus obtained is heated in a dish on a water bath at a temperature not exceeding 140° F., being frequently stirred. The fat being melted the dish is removed from the water bath, and the mass thoroughly stirred so that water and impurities cannot settle on the bottom of the vessel.

The first steps in the examination of the fats are, the determination of water, and of such readily removable foreign substances as adhere to the fats while being produced and have been intentionally or unintentionally added, and the preparation of a pure fat-substance freed from these readily removable matters. It must, however, be borne in mind that a number of fat-like substances, such as resin, paraffin, mineral oils, tar oil, and rosin oil, may be intimately mixed with the fats. These can only be detected and their quantities determined in the further examination of the fats.

*Determination of water.* About 5 grammes of the fat are brought into a tared small beaker or glass dish furnished with a glass rod, and dried, with frequent stirring, till the weight remains constant.

\* *Chemische Technologie*, Bd. 1, S. 160.

Fats, especially tallow, are sometimes fraudulently mixed with caustic potash or potash soap, which imparts to them the property of absorbing larger quantities of water. In this case the fat is not obtained free from water by drying at 212° F., and it is then advisable to determine the content of fat, impurities, and potash, and find the content of water from the difference.

*Determination of non-fats*, i. e., solid foreign substances, such as fragments of skin, parts of plants, dirt, etc. Extract 10 to 20 grammes of fat in a small flask with benzine; then pour through a tared filter previously dried, and wash the latter with the same solvent till no grease spot is left behind when a drop of the filtrate is evaporated upon paper. The dirt contained in the fat is found from the increase in weight of the filtrate.

An abundant organic residue remaining behind in extracting is tested by moistening with iodine solution; the appearance of a blue coloration indicates the presence of amylaceous substances, which is also detected by a microscopical examination of the fat.

Owing to imperfect purification after refining, the fats may contain sulphuric acid, alkaline carbonates, alum and lead. *Sulphuric acid* is detected by vigorous shaking with distilled water, allowing to settle, and compounding the aqueous fluid with barium chloride. A white precipitate indicates the presence of sulphuric acid. A content of *alkaline carbonates* is detected by shaking the oil with water and testing the latter for alkaline reaction with litmus paper. *Alum* is detected by shaking with water to which a small quantity of nitric acid has been added, evaporating the aqueous solution and compounding with ammonia. In the presence of alum a white precipitate is formed.

If a fat contains larger quantities of foreign substances, a direct determination of the content of fat is also made. This may be done in combination with the determination of the content of solid foreign substances by evaporating the filtrate

obtained in a tared vessel, and drying and weighing the residue. In the presence of mucilaginous and amylaceous substances, the determination is more conveniently and more accurately made by mixing about 5 grammes of the fat with four to five times the quantity of finely ground gypsum, drying the mass at 212° F., and then bringing it into an exhauster, the instrument generally known as "Soxhlet's tube" being very suitable for this purpose. An improved form of it, less fragile and more readily constructed, is shown in Fig. 2. The substance to be exhausted is contained in a case of filter paper, which should, however, not be filled up to the top, and to prevent small particles of the substance from being washed away, a piece of cotton-wool is placed upon the substance. To prevent the lower opening of the siphon from being closed by the case, the latter is placed upon an annular strip of sheet-tin. The lower end of the tube *B* is made to pass through a perforated cork into a flask of 100 cubic centimeters' capacity, which has previously been charged with 50 ccm. of solvent—chloroform, ether or benzine. Then bring into the exhausting cylinder enough solvent until it runs off through the siphon, connect *A* with an inverted condenser, and heat the flask on the water-bath. The vapors of the fluid in the flask pass through *B* to *A* and partly also into the condenser where they are condensed. The fluid collects in *A*, penetrates the substance and finally reaches *b*, when it is siphoned off and *A* completely emptied, this process being about twenty to thirty times repeated according to the degree of heating. Filters containing the precipitates to be exhausted are simply folded and placed in the instrument.

The examination of the fats freed from water and non-fats forms the most important part of the analysis. In most cases the admixtures consist only of water and solid substances, and drying and filtering the fatty substance suffice.

FIG. 2.



*Physical methods.* In testing oils it has been sought to utilize the following physical properties: Cohesion, degree of viscosity, specific gravity, melting and congealing points, solubility in glacial acetic acid, and electric conductivity.

*Cohesive figures.* When a drop of oil is allowed to fall gently on the surface of a glass plate or on the level, smooth bottom of a porcelain dish, it assumes different forms. Tomlisson, and later on, Hallwachs, made these forms the subject of special investigation, and found that certain varieties of oil, and even mixtures, assume definite forms by their particles adhering more closely together or spreading out more on the edge, but as yet little success has attended experiments in this direction.

*Viscosity.* The oils, as previously mentioned, are distinguished by a certain degree of viscosity, or resistance to flow, due to the greater or less cohesion between the constituent particles of the liquid. This can be readily determined by the rate at which an oil flows through an orifice of standard dimension.

*Determination of specific gravity.* This is of more importance for testing oils than the preceding methods. Special hydrometers, so-called oleometers or oil-balances, have been constructed for commercial use. They are so weighted or graduated as to adapt themselves to the densities of the leading fixed oils. The instruments in general use are Gobby's and Lefèvre's oleometers, Fisher's oil-balance, and Brix's hydrometer for lighter liquids. The scale of Gobby's oleometer is divided into 50°, and it floats at 0° or zero in pure poppy oil, at 38° or 38.5° in pure almond oil, and at 50° in pure olive oil. The standard temperature of the instruments made in England is now 60° F.; of those made on the Continent, 54.5° F. The oil, before testing it, must therefore be brought to this normal temperature by plunging the glass cylinder containing it into either hot or cold water, as the case may be; or a correction of the observed density must be made. The latter is done by deducting two from the indication of the instrument for each

degree of the thermometer above the normal temperature of the instrument, and adding two for every degree below it. Thus: Suppose the temperature of the oil at the time of the experiment is  $60^{\circ}$  F. and the oleometer indicates  $61^{\circ}$ ; then—

$$60.0^{\circ} \text{ actual temperature.}$$

$$54.5^{\circ} \text{ normal temperature.}$$

$$\underline{5.5^{\circ} \text{ difference.}}$$

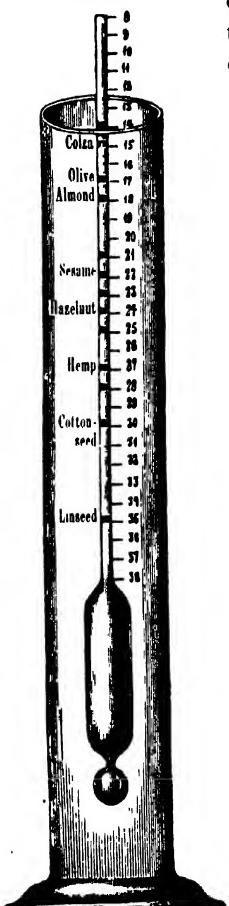
$$\text{Indication of the oleometer } 61.0^{\circ}$$

$$\text{The difference } 5.5 \times 2 = 11.0^{\circ}$$

$$\underline{\underline{\text{Real density, } 50.0^{\circ}}}$$

Lefébvre's oleometer, Fig. 3, is so graduated that the specific gravity is directly read off, when immersed in a fluid at the standard temperature of  $15.5^{\circ}$  C. ( $60^{\circ}$  F.). At various points the average specific gravities of normal oils of different kinds are marked off—linsced oil, cotton-seed oil, etc.—and, to save figures, only the second and third decimal places are given—*i. e.*, 15 indicates 0.9150 specific gravity. To facilitate reading, the oils are indicated by a color as near alike as that acquired by them by the test with sulphuric acid. If the temperature of the oils differs slightly from the normal one of  $15.5^{\circ}$  C., a correction is made by adding (or subtracting)  $\frac{1}{2} \times .001$  for every degree of temperature above (or below) the standard, this correction being based on the fact that most oils expand on heating to nearly the same extent, so that the specific gravity becomes lowered by about .00068 per  $1^{\circ}$  C.

FIG. 3.



Although, generally speaking, oleometers give good results in testing oils as to their purity, the statements cannot be implicitly relied on. The variations in the specific gravities of oils are very slight, and exact experiments have shown that the variations of the specific gravity of one and the same variety of oil are, according to age, mode of production, etc., frequently as great as the difference between the oil and another one used as an adulterant. To overcome this, Laurot, in 1841, constructed an oil-balance for testing the oils at 212° F., but this proved of little service, especially since Scharlach has shown that oils do not expand equally by heating. The determination of the specific gravity as a means of testing fat oils is therefore limited to a few cases, and, generally speaking, cannot be considered reliable. However, the specific gravity may serve as a useful mark of distinction in determining whether two oils are identical or not. According to Donny, one of the two samples is colored slightly red with alkannin, and a drop of it is allowed to fall slowly into the other sample. The drop will be suspended or float upon the surface or sink down; only in the first case the oils may be supposed to be identical.

*Determination of the melting point.*—For this purpose several methods have been proposed, the following being the most important: 1. Suck the fat into capillary tubes and after allowing it to congeal, place the tubes in water. Heat slowly and note by a thermometer dipped into the water the temperature at which the fat becomes transparent. This is the melting point. 2. According to Pohl, the bulb of a thermometer is coated with fat and the thermometer dipped in water. The latter is then heated and the temperature noted at which the fat melts off. 3. According to Bouis, the liquified fat is brought into narrow glass tubes open on both ends. When the fat is congealed the tubes are placed under water, and heat is applied until the liquefied fat is forced by the water, passing in through the lower narrow orifice, to the level of the surrounding layer of water. In all these methods the tubes or

thermometer with the re-congealed fat must be laid aside for one or two days before being used for the experiments, since fats, especially soft ones, acquire only very slowly their natural solidity after having been melted.

In the first method mentioned above, a certain degree of transparency is considered the melting point, and in the others a certain mobility of the particles of fat. That these methods cannot always give correct results is shown by the great variation in the melting points of fats as given by different observers, the statements regarding the melting point of beef-tallow ranging, for instance, between 98.5° and 139° F. While slight differences may, to be sure, be referred to the natural variability of the fats as secretions of living organisms, they cannot be so considerable. Besides some fats, as observed by Wimmel, become entirely transparent only at a temperature a few or several degrees higher than that at which they entirely liquefy, this being the case, for instance, with lard and with beef and mutton tallows.

With the use of capillary tubes, according to the method of Bouis, the fat will rise in them at a temperature which will be the higher the narrower the tubes are. But, even with the use of wider tubes, the results obtained vary according to whether the tubes dip more or less deep into the water, and the temperature of the latter rises more rapidly, or more slowly. According to Rudorff, Pohl's method gives the best results. He coats the bulb of the thermometer with a layer of fat 3 millimeters thick. An agreement as regards an accurate method for the determination of the melting points of fats would be very desirable, and so long as this is not the case, such determinations are of but little value.

There is less uncertainty regarding the melting points of fatty acids separated from a fat or oil. It is therefore preferable to determine in the examination of fats, the melting point of the separated fatty acids.

*Determination of the congealing point.*—The fat is first melted at as low a temperature as possible so that particles of un-

melted fat still float in the portion already melted; it is then allowed to congeal, being constantly shaken. By noting from minute to minute the readings of a thermometer dipped in the fat, it will be seen that with some fats the temperature sinks to a certain degree and after remaining constant for some time sinks still further. As the fat congeals during the constant temperature, the latter must be considered the congealing point. With other fats the temperature sinks to a certain degree while they congeal more and more, but then rises several degrees, whereby the fats become entirely solid. With some of these fats the maximum to which the temperature rises is constant, and is, therefore, to be considered as the congealing point; other fats do not show this constancy, and an exact determination of their congealing points is, therefore, impossible. For the valuation of a fat it is therefore preferable, as in the case of fixing the melting point, to determine the congealing point of the fatty acids separated from it.

The method proposed for this purpose by Dalican has been generally adopted, and is known as the *titre test*. The process is as follows: 100 grammes of the sample are saponified, the separated fatty acids freed from water, and finally filtered through a dry folded filter into a porcelain dish. The fatty acids are allowed to congeal under an exsiccator and stored away over night. The next day the fatty substance is carefully melted, and a sufficient quantity of it is poured into a test-tube, 15 centimeters long and 3.5 centimeters wide, to fill the latter more than half full. The test-tube is then secured by means of a cork in the neck of a flask, 10 centimeters wide and 13 centimeters high, and an accurate thermometer graduated in tenths of a degree is inserted in the fatty acids so that the bulb is in the center of the fatty mass. The latter is then allowed to cool slowly; so soon as a few crystals are observed on the bottom of the test-tube, the mass is stirred with the thermometer, care being had not to touch the sides of the tube, so that all the congealed particles as formed are thoroughly stirred into the mass. The fatty acids thereby become

turbid throughout their entire bulk. The temperature is now accurately noted, it being advisable to make a record in writing of it at certain intervals. At first the temperature falls, then suddenly rises a few tenths of a degree, reaches a maximum, remaining stationary there for a short time, and then falls again. The maximum attained is called the *titre* or congealing point. According to Lewkowitsch this method gives reliable results provided it is always executed exactly under the same conditions.

E. Valenta has recommended the *behavior of fats and fat oils towards glacial acetic acid* as a means of testing them. By experiments he ascertained that most animal and vegetable fats are more or less soluble in glacial acetic acid, but that the behavior of the various oils varies so much that it can be advantageously utilized for their characterization. He made his experiments by intimately mixing in a test-tube equal parts of oil and glacial acetic acid, and exposing the mixture to various temperatures. Of the examined fats dissolved—

1. Completely at an ordinary temperature ( $59^{\circ}$  to  $68^{\circ}$  F.):  
Olive-kernel oil and castor oil.
2. Completely or nearly completely at from  $73.5^{\circ}$  F. to the boiling temperature of the glacial acetic acid: Palm oil, bay oil, expressed nutmeg oil, cocoanut oil, palm-kernel oil, illoopa oil or Mahwah butter, olive oil, cocoa oil or butter, sesame oil, pumpkin-seed oil, almond oil, cotton-seed oil, peanut oil, Himalayan apricot oil, beef tallow, American bone-fat, cod-liver oil.
3. Incompletely at the boiling temperature of glacial acetic acid: Colza oil, rape oil, hedge-radish oil.

Notwithstanding the fact that the temperatures at which the turbidity of the solution of glacial acetic acid commences are subject to considerable variations, chiefly due to the varying percentage of free fatty acids in the fat, this method is very useful for testing oils, especially in connection with others.

Rousseau many years ago made attempts to utilize the *electrical conductivity* of oils for testing them. His method

was based upon the fact that all fat oils, with the exception of olive oil, are good conductors of electricity. He constructed a special apparatus, which he termed "*diagometer*"; but the results obtained were not sufficiently accurate. Rousseau's idea has been recently again taken up by Palmieri, but scarcely with any better results.

*Chemical method.*—The chemical methods employed for the examination of fats are based, 1, upon the fact that oleic acid is converted by nitrous acid into solid elaidic acid, while linoleic acid is not altered; 2, upon the increase of temperature the oils show with concentrated sulphuric acid; 3, upon the fact that the different oils can fix a varying quantity of calcium hydrate; and 4, that they can fix a varying quantity of iodine. For other industries have further to be taken into consideration, the determination of the liquid fatty acids contained in a fat (Reichert-Meissl number); the insoluble fatty acids contained in it (Hehner's number) and the oxy fatty acids and fatty alcohols (acetyl number). Other chemical reactions used refer more to immaterial constituents which the fats have absorbed or retained in their preparation from the vegetable or animal body from which they are derived.

*Elaidin test.*—This test is executed in the most simple manner by bringing a few drops of the oil to be examined upon water in a test-tube, and introducing gas developed by the action of nitric acid upon iron filings. The oleic acid of non-drying oils is converted into a rigid mass, while the drying oils separate, according to their quantity, in drops or as liquid layers upon the surface. The color of the elaidin formed is characteristic for various oils, though it cannot be implicitly relied upon, since there is a possibility of the drying oils not being distinctly separated on account of being enveloped by the non-drying ones.

*The development of heat resulting from the action of concentrated sulphuric acid upon fat oils as a test for their purity* was first proposed by Maumenè. While this method does not suffice for all cases, it cannot be denied that it gives good

results to distinguish, for instance, drying from non-drying oils, the former becoming more heated than the latter. The test is made as follows: Mix weighed quantities of oil and sulphuric acid by stirring with a thermometer and note the maximum rise in temperature. The same test has, under the same conditions, to be made with a typical oil, and the results are then compared.

*Determination of the saponification number i. e. the determination of the quantity of calcium hydrate in milligrammes which is required for the complete saponification of 1 grammme of fat.* This method was first applied by Koettstorfer for the detection of adulterations of cow-butter with other fats. He saponified 1 to 2 grammes of filtered butter fat in a tall, covered beaker of about 70 cubic centimetres capacity with 25 cubic centimeters of alcoholic potash lye of about one-half standard strength, by heating on a water-bath for fifteen minutes and titrating with one-half standard hydrochloric acid, using a very dilute alcoholic solution of phenol-phthalein as an indicator.

According to Valenta, who tested a number of animal and vegetable fats by Koettstorfer's method, the following is the most suitable process: The fat to be examined (if solid) is first melted and then filtered; 1 to 2 grammes are then brought into a wide-necked flask closed, during the subsequent heating, by means of a funnel inserted in the neck.

The standard (titer) of alcoholic potash lye changes very rapidly; it is therefore recommended to test it before every series of experiments. This is done by measuring out 25 cubic centimetres by means of a pipette, then heating ten to fifteen minutes on a water-bath, and finally titrating with half standard hydrochloric acid, using alcoholic solution of phenol-phthalein as an indicator. The lye is prepared by dissolving 28.05 grammes of caustic potash in 96 per cent. alcohol, and increasing the saturated cold solution to 1 liter by the addition of alcohol.

For the determination of the saponification number, the

potash lye is brought to 59° F.; 25 cubic centimetres are then measured out by means of a pipette and added to the quantity of fat in the flask. The whole is then heated on a water-bath kept near the boiling temperature. Heating for ten to fifteen minutes generally suffices, though some fats require a longer time; for cocoanut-oil it is, however, recommended not to exceed twelve minutes in order to obtain useful results. The clear soap solution is compounded with phenolphthalein, and the excess of standard potash titrated back with half standard hydrochloric acid, the point of neutrality being very sharply indicated by the yellow color of the liquid.

The final results are expressed by the number of milligrammes of potash (KHO) which saponify 1 gramme of fat.

According to the results obtained, Valenta divides the oils into three groups:—

1. Mean saponification number, 193.0: Oils of apricot, almond, peanut, cotton-seed, olive, and sesame.
2. Mean saponification number, 188.1: Oils of pumpkin-seed, olive kernels.
3. Mean saponification number, 177.1: Oils of colza, rape, hedge radish, and castor.

*Iodine absorption process.*—This process recommended by Baron Hübl is of great value for testing fats. The non-saturated fatty acids are capable of combining in their free state, as well as in the form of their glycerides, with halogens; each molecule of oleic acid and its homologues absorb 2 atoms of chlorine, bromine or iodine, that of linoleic acid 4 atoms, and that of linolenic acid 6 atoms.

Of the haloids the use of iodine was for many reasons found more convenient and suitable than chlorine or bromine. Experiments, however, soon showed that at an ordinary temperature iodine acts very inertly upon fats, while at higher temperatures its effect is very unequal, and a smooth reaction in the above-indicated sense cannot under these circumstances be produced. An effect satisfactory in every respect is, however, produced by an alcoholic solution of iodine and mercuric

chloride. This mixture reacts upon the non-saturated fatty acids at an ordinary temperature, products of chlorine and iodine-absorption products being formed, while the saturated acids present remain unaltered. The mixture acts in the same manner upon the free fatty acids, as upon glycerides, which, together with the easy volumetric determination of iodine, makes this test a very simply one. Hence for the determination of the quantity of iodine which a fat may absorb, a weighed sample of it has to be treated with an excess of a measured quantity of the alcoholic solution of iodine and mercuric chloride, diluting, after reaction is complete, with water, and to determine volumetrically the iodine present, adding potassium iodide. From a practical stand-point it makes no difference whether only iodine has entered into combination, or iodine and chlorine, because in the volumetric determination under the above mentioned conditions both elements are equivalent. Experiments have shown that in order to exhaust the action of the total iodine, at least 1 molecule of mercuric choride is required for every 2 atoms of iodine. Since most fats dissolve with difficulty in alcohol it is advisable, in order to facilitate reaction, to add chloroform which is entirely indifferent towards iodine solution.

The alcoholic solution of iodine and mercuric chloride possesses the disagreeable feature of not being very constant, and it is therefore necessary to test the solution previous to every series of experiments.

For the execution of the process are required : Iodine and mercuric chloride, sodium hyposulphite solution, chloroform, potassium iodide solution, and starch solution. The aleoholic solution of mercuric chloride and iodine is prepared by dissolving, on the one hand, 26 grammes of iodine in 500 cubic centimeters of 95 per cent. alcohol free from fused oil and, on the other, 30 grammes of mercuric chloride in 500 ccm. of alcohol. The latter solution, if required, is filtered, and both solutions are then combined. The liquid should be allowed to stand 6 to 12 hours before use. For the sake of

simplicity this solution will in the following discussion be simply called iodine solution.

For the sodium hyposulphite solution it is best to use about 24 grammes of the salt in 1 liter of water. It is standardized with pure sublimed iodine. The solution may be considered stable except for extremely accurate determinations, which is here, however, by no means the case.

The chloroform, before use, has to be tested as to its purity. For this purpose mix about 10 ccm. of it with the same quantity of the iodine solution, and after two or three hours volumetrically determine the quantities of iodine in this fluid as well as in 10 ccm. of the stock solution. The chloroform may be used if exactly corresponding numbers are obtained in both cases.—The potassium iodide solution is an aqueous solution in the proportion of 1:10.—The starch solution is fresh 1 per cent. paste.

The fat is preferably weighed in a light small glass. After melting the fat is emptied into a flask having a capacity of 200 ccm. and furnished with a glass-stopper, and the glass together with the fat still adhering to it is again weighed. The quantity of the sample depends on the probable absorption of iodine, 0.2 to 0.3 gramme of drying oil being as a rule used, 0.3 to 0.4 gramme of non-drying oil, and 0.8 to 1 gramme of solid fats. The fat is dissolved in about 10 ccm. of chloroform and 20 ccm. of iodine solution are added. Should the fluid not be perfectly clear after shaking, add a little more chloroform. If after standing a while the color of the fluid lightens materially, it is an indication of not sufficient iodine solution having been used, and about 5 to 10 ccm. more of it are added by means of a pipette. Enough iodine solution must be used for the fluid to show a deep brown coloration after standing for 1½ to 2 hours. Reaction is complete after this time, and the quantity of free iodine is then determined. For this purpose the product of the reaction is compounded with 10 to 15 ccm. of potassium iodide solution, shaken, and diluted with about 150 ccm. of water. A portion of the iodine

is contained in the aqueous solution, and another in the chloroform which in diluting has separated and dissolved the iodized oil. Sodium hyposulphite solution is now allowed to run in, whilst shaking frequently, from a burette graduated in 0.1 ccm., until the aqueous fluid, as well as the layer of chloroform, shows only a slight coloration. Now add a little starch solution and finish the operation by carefully adding sodium hyposulphite solution and frequently shaking the closed flask. Immediately before or after the operation, 10 or 20 ccm. of the iodine solution are titrated with an addition of potassium iodide and starch solution. Taking into consideration the strength of the sodium hyposulphite solution, the quantity of iodine fixed by the fat is found from the differences of these two determinations. The quantity of iodine found is stated in per cents. of the fat and this number is termed the *iodine number*. The numbers are quite constant provided a sufficient excess of iodine solution has been present; this excess should, according to Benedikt, amount to not less than 30 per cent. of the quantity of iodine used. The result is independent of the concentration and an excess of mercuric chloride, and at least 1 molecule of the latter must be present for 2 atoms of iodine. According to Hübl it does not matter whether titration is effected after standing for 2 or 48 hours, but so as to be on the safe side, it should be executed only after 4 to 6 hours.

Other chemical reactions used in testing fats consist of color phenomena appearing under the action of certain chemicals. Mailho has, for instance, observed that all oils from Cruciferæ (rape, colza, German sesame) form, when treated with aqueous potash or soda lye, potassium sulphide and sodium sulphide, but that this formation does not take place in other oils. He boiled 25 to 30 grammes of the oil with a solution of 2 grammes of pure sodium hydrate in 20 grammes of water and then filtered through a filter previously moistened. A strip of paper previously moistened with solution of lead acetate or silver nitrate becomes black if the oil is derived from Cruciferæ or contains such in admixture. If the sample is

boiled in a silver dish, the latter becomes black, even if the oil under examination contains but one per cent. of an oil derived from the Cruciferæ. This manipulation is, however, liable to errors, since oils extracted with carbon disulphide are generally not entirely free from sulphur combinations, and therefore give the same reaction. Further, it occurs sometimes that barrels used for transporting oils are rinsed with sulphurous lyes, and the oil, though pure, will show the same reaction. To avoid this error it suffices to wash the oil, before testing, with pure water, stirring it with a silver spatula, whereby the alkaline sulphide, which is soluble in water, blackens the silver. The washing is continued until the water shows no trace of sulphur. The oil can then be tested in the above manner.

Schneider's method of testing oils for adulteration with colza or rape-seed oils may be considered as a modification of Mailho's. He shakes the oil with double its volume of ether, adds 20 to 30 drops of a saturated solution of nitrate of silver, and observes the coloration of the oil. If it contains colza or rape-seed oil, it becomes brown to black.

Th. Chateau\* and C. Calvert† have compiled extensive tables of chemical characteristics for the various fluid fats. However, as the color reactions appearing by the action of chemical agents readily suffer alterations by the modes of gaining and purifying the oils, as well as by their age, they are of doubtful value, and these tables are therefore omitted.

*Determination of rosin oils and mineral oils.* The fat oils are frequently adulterated with cheaper fat oils, principally rosin oils and mineral oils. The most reliable method of showing their presence is the saponification process first recommended by Thompson, *i. e.*, saponifying the oil under examination, mixing the soap formed with sand, drying the soap, and extracting with benzine. The method proposed to shake the

\* Th. Chateau. *Die Fette*, Leipzig, 1864.

† Dinglers's Polytech. Journal 132, S. 282.

liquid soap directly with benzine and to separate the latter from the soap solution by means of a separatory funnel, cannot be recommended. Finkener\* declares the last method entirely useless when the oil contains less than 10 per cent. of non-saponifiable oil, and uses alcoholic soda lye for saponification, while Thompson recommended alcoholic potash lye. Finkener prefers soda, because potash-soap is more difficult to dry than soda-soap, and proceeds as follows:

Dissolve 35 grammes of *Natrium hydricum purum* in 85 cubic centimetres of water and pour the hot solution into 730 grammes of boiling hot alcohol. Bring 10 grammes of the oil to be examined into a flask of 300 cubic centimetres' capacity, add 50 cubic centimetres of the alcoholic soda solution, boil the whole for 15 minutes on a water-bath and compound with 5 grammes of sodium bicarbonate for the conversion of the excess of soda used into carbonate. Pour the solution upon 200 grammes of pure dry sand in a metal evaporating dish and heat on a water-bath, stirring constantly, until the odor of alcohol has entirely disappeared. The warm mass is then brought into a glass cylinder of 500 cubic centimetres' capacity and provided with a stopper, and, after cooling, 300 cubic centimetres of benzine, with a boiling point below 212° F., are added and the whole is thoroughly shaken for some time. The benzine is then filtered off through a dry filter into a dry flask and 150 cubic centimetres of the filtrate are distilled in a still. The residue from the still is brought with a little benzine upon a watch-crystal and dried on a water-bath until the benzine disappears. The residue is derived from 5 grammes of oil.

In examining commercial oils in the manner described, 0.5 to 3 per cent. of non-saponifiable residue is obtained even if there be no reason for suspecting adulteration. An intentional addition of mineral-oil can, therefore, be only assumed when more than 5 per cent. of non-saponifiable matter is found.

\**Mittheilungen aus der Koenigl. techn. Versuchsanstalt, Berlin, 1886, S. 13.*

Whether the residue consists of rosin-oil or mineral-oil is recognized by treating it with a mixture of 10 volumes of alcohol of 0.8182 specific gravity at 60° F. and 1 volume of chloroform. Rosin oils dissolve, according to Finkener's\* investigations, at 73.5° F. by shaking with 10 times their volume of this mixture, while mineral oils with a high boiling point do not dissolve even if shaken with 100 times their volume. Demski and Morawski† treat the oily residue with acetone. If it dissolves in an equal volume of acetone, rosin-oil, or rosin-oil compounded with a little mineral-oil, is indicated; if a portion remains undissolved, the sample consists of mineral-oil or mineral-oil compounded with a little rosin-oil.

The presence of *rosin* in fats is readily shown by its solubility in alcohol and soda solution. By repeatedly heating the sample of fat with 70 per cent. alcohol, the rosin passes into solution. After precipitating it with water, the precipitate is concentrated by heating, and, if necessary, by the addition of some hydrochloric acid; it can then be readily recognized as rosin by its appearance, odor, etc. Barsfoed heats the fat with a soda solution prepared by dissolving 1 part of crystallized soda in 3 parts of water and adding 7 parts of 30 per cent. alcohol (2 volumes of alcohol of 93 per cent. and 5 volumes of water). The rosin passes into solution and is separated by acidulating and heating.

As the commercial fats contain more or less free fatty acids, the above methods are not available for the quantitative determination of rosin. For this purpose the fat must first be saponified and the quantity of rosin determined as described later on for the determination of rosin in soaps.

The presence of *free fatty acids* in oils can be readily detected. A very simple method is given by Wiederhold.‡ By pouring the oil to be examined over cuprous oxide in a white

\* Mittheilungen aus der Koenigl. techn. Versuchsanstalt, Berlin, 1885, S. 160.

† Dingler's Polytechn. Jour. 258, S. 39.

‡ Dingler's Polytechn. Journ., 217, 314.

glass, the layer next to the cuprous oxide assumes a green color if the oil contains acid. The appearance of the reaction is promoted by moderate heating. To test the neutrality of the oils Allaire\* shakes them with a solution of sodium bicarbonate in water. If the oil separates in shining globules it is neutral, but if it becomes turbid and a partial saponification takes place it contains free fatty acids.

For the quantitative determination of fatty acids, Mayer dissolves 2 to 3 grammes of fat in 20 cubic centimetres of ether, compounds the solution with 10 cubic centimetres of alcohol and some solution of phenolphthalein, and adds, according to the content of acid,  $\frac{1}{10}$  or  $\frac{1}{4}$  standard lye, until the liquid assumes a red coloration. Aqueous potash lye is preferable to the alcoholic, on account of the constancy of its strength, which constantly changes in the alcoholic solution, and must, therefore, always be determined anew before each series of experiments; on the other hand, the fat readily separates from its alcoholic solution by the addition of aqueous lye, and has then again to be brought into solution by heating on a water-bath. With an alcohol-ether solution two layers are formed, and the reaction must be carried on to the end, shaking vigorously after each fresh addition of lye.

The quantity of calcium hydrate in  $\frac{1}{10}$  per cent. of the number of milligrammes of it required for the neutralization of the free fatty acids contained in 1 gramme of fat is termed the *acid number*. Hence it forms a measure for the content of free fatty acids in fat.

When a fat or oil consists exclusively of neutral glycerides the acid number is of course equal 0. In this case the saponification number indicates the quantity of calcium hydrate which is required for the saponification of the neutral esters, or in other words, the quantity of calcium hydrate which is required for the neutralization of the fixed fatty acids. When the fats or oils contain free fatty acids, hence possess an actual

\* Octave Allaire, Notice sur les huiles neutres raffinées, p. 11.

acid number, then their saponification number represents the total of the quantities of calcium hydrate which are required for the neutralization of the free fatty acids, as well as of those fixed on glycerin. The difference between these two quantities of calcium hydrate is called the *ester number*. Hence, by it is understood the number of milligrammes of calcium hydrate which are required for the saponification of the neutral esters in 1 grammie of fat or oil.

Since the introduction of the so-called carbonate saponification the determination of the content of neutral fat in fatty acid has become of great importance for the soap manufacturer. If the saponification number of the fat from which the fatty acid has been obtained, is known, which is possible when the latter has been prepared by the soap manufacturer himself, the content of neutral fat is readily found by determining the saponification number and the acid number of the fatty acid. If the saponification number of the fatty acid found  $V = 202$ , the acid number  $S = 162$ , then the difference  $V - S = 202 - 162 = 40$  corresponds to the neutral fats present in the sample. If the saponification number of the saponified fat is 195, the following proportion results :

$$195 : 100 = 40 : X = 20.05.$$

Hence the fatty acid contains 20.05 per cent. of neutral fat and  $100 - 20.05 = 79.95$  per cent. fatty acid.

If the saponification number of the fat used is not known, which is always the case with bought fat, proceed as follows: Pour hot alcohol over a few grammes of the sample, add phenolphthalein, and carefully neutralize the fatty acids by allowing to run in from a burette dilute lye until the fluid acquires a permanent red coloration. Allow the fluid to cool, and then dilute with an equal volume of water. Next bring the fluid into a separatory funnel and shake it out with benzine or ether. When allowed to rest, two layers are formed, the lower one containing the aqueous soap solution and the upper one the ethereal fat solution. Draw off the soap solution into another separatory funnel and shake it out with

fresh ether. Then combine the two ethereal solutions, wash the fluid with a small quantity of water to free it from traces of dissolved soap and transfer it to a tared flask. Evaporate the ether on a water bath, dry the residue at 212° F., and weigh.

*Determination of glycerin.*—The quantity of glycerin contained in a fat is now generally determined by calculation from the saponification number or from the ester number. In the saponification of a fat three molecules of calcium hydrate are equivalent to 1 molecule of glycerin. Hence 168 grammes of calcium hydrate are equal to 92 grammes of glycerin, or 1 gramme calcium hydrate to 0.5476 gramme. Hence the ester number (d) consequently the difference between saponification number and acid number has to be determined, and we have then  $g = 0.5476 d$ .

Another method very much in use for the determination of the glycerin consists in saponifying the fat with alcoholic potash or soda lye, expelling the alcohol by evaporation, dissolving the soap in water, adding dilute sulphuric acid and boiling moderately until the fatty acids have become entirely clear. The mass is then allowed to cool, and, after filtering the liquid containing glycerin from the congealed fatty acids, the latter are once more boiled in water, allowed to congeal, and the filtered wash-water is united with the first filtrate. The latter is exactly neutralized with sodium carbonate and then evaporated to dryness on a water-bath. The residue, consisting of sodium sulphate and glycerin, is treated with alcohol, which leaves the sodium sulphate undissolved. The filtered alcoholic solution is evaporated, the residue again treated with alcohol, and the filtered solution evaporated in a platinum dish on a water-bath.

On account of the volatility of glycerin at 212° F., the numbers obtained by the foregoing process are always too low. The most reliable process, and one which is available for all cases, is the determination of the glycerin by oxidation with potassium permanganate.

When glycerin in a strongly alkaline solution is oxidized at an ordinary temperature with potassium permanganate, 1 molecule of glycerin yields quantitatively exactly 1 molecule each of oxalic and carbonic acids—



Upon this is based the determination of glycerin by Benedict and Zsigmondi,\* the principle having been first established by Fox.

The fat is saponified with calcium hydrate and perfectly pure methyl alcohol, the residue dissolved in hot water, and the soap decomposed with dilute hydrochloric acid. It is then heated until the fatty acids have separated clear. With fluid fats it is advisable to add some hard paraffin in order to congeal the fatty acids floating on top during the subsequent cooling, which is effected by placing the dish in cold water. The fluid is then filtered into a capacious flask, neutralized with potash lye, and 10 grammes of caustic potash are added. 5 per cent. solution of potassium permanganate is then added at an ordinary temperature until the fluid is no longer green, but assumes a blue or blackish coloration. It is then heated to the boiling point, whereby manganic peroxide is separated and the fluid becomes red; as much aqueous sulphurous acid is then added as is required for complete decoloration. It is then filtered through a smooth filter of sufficient size to receive at least one-half of the entire fluid, and thoroughly washed with boiling water. The last wash-waters are frequently rendered turbid by some manganic peroxide; this turbidity disappears, however, by the action of the sulphurous acid liberated during the subsequent acidulation with acetic acid. The fluid is then heated to nearly the boiling point and precipitated with calcium chloride or calcium acetate. As, besides calcium oxalate, the precipitate contains always some silicic acid and frequently gypsum, it cannot be considered, after calcining, as pure calcium carbonate, nor as

\* *Chemiker Zeitung*, 9, S. 975.

calcium oxide. The determination of the calcium oxalate contained therein is best effected by titration, either with potassium permanganate in acid solution or by alkalimetry after calcining. For the latter purpose the calcined precipitate is dissolved in about half-standard solution of hydrochloric acid, titrated back with about half-standard soda lye with an addition of dimethyl-aniline orange as an indicator. The titer of the hydrochloric acid is generally fixed for sodium carbonate; 106 parts of sodium carbonate correspond to 92 parts of glycerin.

In connection with the above process it may be mentioned that methyl alcohol is used for the saponification of the fats instead of ethyl alcohol, because the latter, at certain concentrations and with a determined percentage of alkali in the solution, is converted by potassium permanganate into oxalic acid. This creates errors which are the greater the more alcohol is retained by the soap in drying. By repeated evaporation and renewing of the water to expel the last traces of alcohol, a portion of the glycerin would also be lost.

The fluid subjected to oxidation contains besides glycerin all the soluble fatty acids; by oxidation with potassium permanganate, according to the above process, they yield, however, neither oxalic acid nor another acid which can be precipitated by lime in acetic acid solution, so that their presence does not influence the determination of the glycerin.

Whether an oil obtained by extraction is free from carbon disulphide can, according to O. Braun, be readily recognized by stirring about 30 parts by weight of lye of 40° B. under 60 parts by weight of oil, and allowing the soap formed to stand in a warm place for one hour. If the oil is badly purified, the soap is dark green and of a bad odor, which, however, disappears in time (weeks or months), leaving the soap externally faultless.

## CHAPTER V.

### FATS, FAT OILS, FATTY ACIDS AND ROSIN USED IN THE MANUFACTURE OF SOAP.

THE fats are generally divided, according to their derivation, into animal and vegetable fats.

#### I. ANIMAL FATS.

*Tallow* is the generic term for the rendered fat of cattle and sheep. *Beef tallow* includes that from oxen, cows and calves, and *mutton tallow* that from sheep and goats. The terms *tallow* and *suet* are often used indiscriminately to denote the adipose tissue of the ox and sheep. However, the term *suet* should only be applied to the untreated animal fatty tissues, whilst the word *tallow* should only imply the fatty matters thence extracted and freed from cell walls, etc. In this sense *tallow* includes the rendered fat obtained from the ox, sheep, goat, stag and other quadrupeds, including the horse and hog, the fat of the former being generally known as horse-grease and that of the latter as lard. The crude fat is enveloped in very thin cellular tissues, and more or less contaminated with particles of skin, blood, etc. If kept for several days these particles become decomposed and putrefy. The crude fat should therefore be kept in a cold place or at once separated from the tissues by the process known as *rendering*. This is effected either in open kettles at a low temperature or by steam under pressure in closed tanks, the latter process being employed for all kinds of stock unsuitable for food products. The roasted tissues from the process of rendering in open kettles are known as *greaves or scrap*, and are used for feeding pigs and poultry; the tissues from the steam process, which

have not been subjected to so great a heat, are utilized for making ammonia and glue.

Dilute sulphuric acid is sometimes used for disintegrating the membranes enclosing the fat. Bring 50 pounds of dilute sulphuric acid into the kettle, then add gradually 1000 pounds of the comminuted fat divided into four equal portions, and finally 150 pounds of water previously compounded with 5 pounds of sulphuric acid of 60° B. The mass is then heated. By the action of the sulphuric acid, which partly dissolves and partly decomposes the membranes, the process of rendering even large quantities of tallow is finished in 1½ to 2½ hours. D'Arct originally proposed the addition of acid only for rendering over an open fire, but it is also available for steam rendering, which is now employed in all large establishments. In most rendering establishments, not working with a closed apparatus, open wooden vats lined with lead, and direct steam are employed. For 100 pounds of tallow, 20 pounds of water and 1 pound of sulphuric acid of 66° B. are used, and steam of a pressure of 1 to 2 atmospheres is introduced. Another very suitable process consists in pouring sulphuric acid of 4° to 5° B. over the crude tallow as delivered by the butchers and loading it with boards and stones so that the sulphuric acid stands over the fat. After four or five days the acid is drawn off through a faucet in the bottom of the vat. The tallow is then rendered by the introduction of direct steam, the process being quickly accomplished, since the cells have been partly dissolved by the acid. The greaves, which still contain tallow, are again acidulated and rendered out.

Evrard has proposed to mix and warm 300 parts of comminuted tallow with caustic soda lye (made of 1 part of soda ash dissolved in 200 parts of water). The odorous substances combine with the soda and remain in the lye dissolved, while the pure fat is separated. Though the Société d'Encouragement of Paris examined this process and declared it an improvement, rendering with sulphuric acid deserves the preference.

Before the introduction of palm oil, cocoanut oil, and palm kernel oil, and the use of beef fat for the manufacture of oleomargarine and other food products, tallow was the most important raw material employed in the soap industry. At present only inferior grades reach the soap manufacturer, the selected tallow, such as the caul fat of beef being used for what is known as *oleo-stearin* and *oleo-oil*. For this purpose the fat is freed from adhering particles of flesh and blood by soaking in warm water. It is then chilled and hardened in ice water, after which it is introduced into a hashing machine and, when thoroughly disintegrated, melted in steam-jacketed kettles at about 150° to 160° F., being constantly agitated by a suitable stirring apparatus. The melted fat is then allowed to stand for the floating particles of membrane or scrap to settle, the process being promoted by the addition of common salt. The fat freed from scrap is drawn into vats and allowed to stand at a temperature of about 95° F. for the crystallization of the stearin, a layer of the latter forming on the top and another on the bottom, with the clear oil between them. The mass thus formed is then thoroughly mixed by stirring, wrapped in small packages of canvas, and gradually subjected to strong pressure. The fat pressed out forms what is known as *oleo-oil*, and is used in the manufacture of oleomargarine, while the stearin remaining as a solid cake in the press cloth is employed in connection with cottonseed oil in the manufacture of *lard compound*.

By subjecting tallow to pressure at a low temperature, *tallow oil* is obtained, which remains liquid at the ordinary temperature.

Commercial tallow frequently has a dirty gray color. Such tallow, to be worked into stearin by saponification, needs refining, as otherwise candles manufactured from it will not show the white appearance so highly valued. The refining consists in remelting the tallow with water, generally with an addition of common salt, alum, or soda.

A very simple method to bleach tallow is to melt it and

then stir in 6 to 10 pounds of soda lye of 20° to 24° B. for each 100 pounds of tallow, and allow it to stand and settle. The brown sediment can be used for rosined grained soaps.

In a pure state the tallows are nearly inodorous; on exposure to the air for some time, mutton tallow, however, acquires a peculiar odor, which, according to Chevreul, is due to the evolution of a volatile fatty acid which he terms *hircic acid*, the existence of which is, however, doubtful.

Tallow consists of stearin, palmitin, and olein. Their proportions vary in the fat from the different parts of the animal, the pouch-fat being richest in olein, and that of the intestines richest in stearin.

There is no great difference in the saponification numbers of the different varieties of tallow, which is readily explained by the fact that oleic acid and stearic acid fix nearly the same quantities of alkali, 1 grammie of oleic acid requiring 198.7 milligrammes of caustic alkali for saponification, 1 grammie of palmitic acid 218.9 milligrammes, and 1 grammie of stearic acid 197.3 milligrammes.

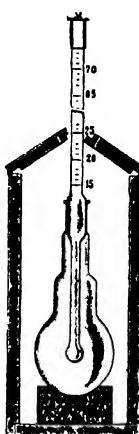
The iodine number of tallow is 40, according to Hübl, and that of the fatty acids separated from tallow 25.9 to 32.8, according to Demski and Morawski.

In commerce, tallow, independent of its color, is now generally valued by the so-called *tallow titre*, *i. e.*, according to the congealing point of its fatty acids. For its determination, Finkener recommends the apparatus shown in Fig. 4, which is also used by the German custom officers. It consists of a four-cornered beechwood box with sides 0.35 inch thick, and having a width of 27.55 inches in the clear and a depth of 56.98 inches. It contains a glass flask having a bulb 1.92 to 2 inches in diameter. A thermometer is ground into the neck of the flask. The flask stands in a shallow cavity of a piece of cork 0.86 inch thick secured to the bottom of the box. When the thermometer is fixed in the neck of the flask the center of its bulb coincides with that of the flask. In the ground part of the thermometer, parallel to the axis, is a

groove, so that the air in the flask always stands under the pressure of the atmosphere. The lower end of the neck of the flask is somewhat enlarged (0.98 inch), so that when the flask has been filled with liquid fat to the mark on the neck, the bulb of the flask remains filled when the fat congeals. The bulb of the thermometer is 0.35 inch in diameter, the thinner portion of it 0.19 inch, and the ground part 0.47 inch. The thermometer is graduated up to 75° C. (167° F.), and can be read in  $\frac{1}{5}$  of degrees. The tube of the thermometer has a somewhat larger reservoir, so that the thermometer can be heated up to 100° C. (212° F.) without bursting. The determination of the congealing point is effected as follows: An average sample of the fat is saponified with alcoholic potash lye in the manner previously described. Of the separated, washed, and dried fatty acids 150 grammes are melted in an uncovered porcelain dish on a boiling water-bath, and then allowed to stand at least ten minutes until the fat forms a clear fluid. The flask is then filled with fat up to the mark on the neck. It is then immediately placed in the box and the lid of the latter closed to prevent rapid cooling. When the thermometer has fallen to about 50° C. the temperature is every two minutes read off and noted. After some time the thermometer falls more slowly, remains stationary for a few minutes, rises again, and finally commences again to fall. The highest reading indicates the congealing point. When the determination has been finished the flask is placed in warm water, the melted fat poured out, and the cold flask cleaned with ether.

Tallow, as found in commerce, is frequently contaminated with water, dirt, particles of skin, etc. Such contaminations are readily detected by the method given on page 55. It is

Fig. 4.



further adulterated with cheaper fats, such as bone fat, kitchen fat, residue from the manufacture of margarin, etc. Such adulterations cannot always be detected with certainty. The best guide is the determination of the melting point, or, still better, that of the fatty acids separated from the tallow, which, according to Dalican, should never be below 111° F.

Tallow adulterated with distilled wool fat has also made its appearance in commerce. This adulteration can, however, be readily detected by the considerable percentage of cholesterol in the wool fat. Saponify the suspected tallow with caustic potash and shake the resulting soap with ether. The latter absorbs the cholesterol and leaves it behind on evaporation. By adding hydrochloric acid and chloride of iron to the residue a violet coloration is produced.

Adulteration with cotton-seed stearin is detected by melting the fat, crystallizing it in a drying box at 95° F., after eighteen hours pressing it through a cloth, and determining from the filtrate the iodine number, which, with filtrate from tallow containing cotton-seed oil, is from 75 to 80, and with that from pure tallow about 55.

Tallow, when used by itself, only saponifies with weak lyes, requiring, according to its age, in the commencement of the operation a lye of 8° to 10° B. With this it readily forms an emulsion, which on reaching the boiling point is converted into a chemical combination. By continued boiling with this lye a viscid, thick soup-paste is obtained, such as is not the case with any other fat. However, boiling, as a rule, is not continued with such weak lye, lye of 12° to 15° B. being used until the combination forms a clear viscid paste, the exact fitting of which is recognized by a gray edge which should rapidly form upon the spatula.

After the introduction of the combination with lye, most fats take up the further lye required very rapidly and well; with tallow this process, however, takes place very gradually, and it is, therefore, necessary to add the lye in portions. By adding the portions of lye in rapid succession, it may happen

that the soap-paste, notwithstanding it shows sharpness, is turbid and not saturated. This defect is best remedied by the addition of some weak lye with a moderate fire; the paste gradually clarifies until all sharpness disappears, and only when this is the case will it be possible to determine how much lye is required for complete saponification. It may further occur that by adding too concentrated lye (of about 20° B. or more) the combination formed may be entirely destroyed so that lye runs off. This fault is remedied with weak lye or water and boiling slowly. In very difficult cases it is best to interrupt boiling and squirt water over the soap-paste.

Saponification is more readily and better effected by direct steam than by an open fire, since a large quantity of tallow can in a short time be converted into a clear paste free from scum, which, when properly "salted out," also yields a finished grain free from scum, so that for most purposes clear boiling is not required.

100 pounds of tallow saponified with soda lye yield at the utmost 165 lbs. of soap, but with so large a yield the product possesses but little solidity and contains water.

*Lard* is the collective term applied to the fat of the hog, and according to the mode of rendering is classified as kettle and steam. The fat varies according to the part of the body from which it is taken, and from the material used the following classification may be made.

1. *Neutral lard* is composed of the leaf of the slaughtered animal in a perfectly fresh state. The leaf is either chilled in a cold atmosphere or treated with cold water to remove the heat. It is then reduced to a pulp in a grinder and passed at once to the rendering kettle. The fat is rendered at a temperature of 105° to 120° F. Only a part of the lard is separated at this temperature and the rest is sent to other rendering tanks to be made into another kind of product. The lard obtained as above is washed in a melted state with water containing a trace of sodium carbonate, sodium chloride or a

dilute acid. The lard thus formed is nearly neutral and is almost exclusively used in the manufacture of butterine.

2. *Leaf lard.* The residue unrendered in the above process is subjected to steam heat under pressure, and the fat thus obtained is leaf lard.

3. *Choice lard.* The remaining portions of the leaf, together with the fat cut from the backs, are rendered in steam-jacketed open kettles under pressure, and produce a variety of lard known as "kettle-rendered." The hide is removed from the back fat before rendering, and both leaf and back fat are passed through a pulping machine before they enter the kettle.

4. *Prime steam lard.* The whole head of the hog, after removal of the jowl, is used for rendering. The heads are placed in the bottom of the rendering tank. Any fat that may be attached to the heart of the animal is also used. The fat is pulled off the small intestines and also placed in the tank. In houses where kettle-rendered lard is not made, the back fat and trimmings are also used. When there is no demand for leaf lard, the leaf is also put in the rendering tank with the other portions of the body mentioned. It is thus seen that prime steam lard may be taken to represent the fat of the whole animal or only portions thereof.

Lard is rendered in a similar manner as tallow. It is of a granular salve-like consistency, has a pure white color and an agreeable taste. On exposure to air it soon turns yellow and rancid. It consists of the glycerides of lauric, myristic, palmitic and oleic acids, as well as small quantities of linoleic acid and, perhaps also, of linolenic acid. The statements regarding the melting point of lard range from 79° to 105° F. Independent of the different methods employed in the determinations, these variations may be largely due to the varying solidity of the fat from the different parts of the body of the animal. The melted lard congeals very slowly, and only after considerable time reacquires its original solidity. The saponification number of lard is, according to Valenta, 195.3 to 196.6, and its iodine number, according to Hübl, 57.6 to 60.

The products known as *lard oil* and *lard stearin* are obtained from lard in the same manner as oleo-oil and oleo-stearin from tallow. Lard oil is used as a lubricant, for illuminating purposes, and in the wool industry. Lard stearin is employed in the manufacture of lard compound, and inferior qualities of it as soap stock.

Lard is frequently adulterated, the principal sophistication being with water; alum or lime is sometimes added to enable it to absorb large quantities of water. An addition of 1 per cent. of slaked lime or of 2 to 3 per cent. of alum will cover 10 to 12 per cent. of water. By melting and recongealing the lard, the foreign substances form a deposit on the bottom and can be readily determined.

The behavior of lard towards lye is similar to that of tallow; the older and more rancid it is the quicker saponification is accomplished, while the fresh neutral fat requires in the commencement a weak lye. Lard is much liked for the preparation of smooth, white grained soaps. Though lard by itself gives a very white soap, even if by bad treatment it has acquired a yellowish or dirty gray color, it is, on account of the lardaceous and somewhat soft consistency of the soap, better adapted for working in connection with palm-kernel oil and cocoanut-oil, which give lean and brittle soaps, an addition of one-third or two-fifths of lard being sufficient in order to obtain a beautiful, solid, and delicate soap. Good lard is at present used only for toilet soaps.

The lard from animals that have died by sickness has generally a dark color and bad odor. In connection with palm-kernel oil it yields good results; the best proportions are 2 parts of lard to 3 of palm-kernel oil or 3 parts of lard to 5 of oil. A larger quantity of lard might yield a soft or mottled product.

The yield of pure ground or fitted grained soap will generally reach about 155 per cent., though the softer a fat the lower the yield of pure grained soap.

*Horse grease.* Generally speaking, the horse is poor in fat.

The careless manner in which the grease is prepared from the carcass renders it rather repulsive, yet, when extracted from the recently slaughtered animal, it constitutes a very suitable and good material for the manufacture of soap. Horse fat varies in consistency according to the organs from which it is taken and the care given to its production. It is either solid, forming a real tallow, or is more or less of the consistency of lard. The fat found in commerce has generally a more or less dark color, a bad odor, and the consistency of lard. As a rule it is not pure horse fat, but contains lard, bone fat, etc. By treating with strong lye in a similar manner as described for lard, it can be bleached, and forms then a very good material for white soaps, while the dark soots can be utilized for dark soaps. Most of the fat being quite rancid, combination is effected with a medium lye, complete saponification taking place with ease and rapidity. The application of this fat for household soaps is about the same as that of lard. Its peculiar sweetish odor makes it a suitable addition to palm-kernel oil to weaken the strong odor of the soap prepared from the latter.

*Bone fat.*—The bones of all animals contain fat. Its constitution appears to be about the same as that of other animal fats, it being only somewhat richer in the glyceride of oleic acid and is, therefore, softer and melts with greater ease. The fat, or at least a considerable portion of it, can be obtained by bringing the crushed bones into a kettle partly filled with water and heating to the boiling point. The fat floats on the surface and is skimmed off with a shallow iron ladle. It is separated from the solid particles by passing through a strainer. When no more fat separates the bones are taken from the kettle by means of a large perforated shovel and replaced by fresh ones so that the same water may be used several times.

Most bone-fat is now obtained as a by-product in the manufacture of animal charcoal or bone-meal. The fat is extracted either by steaming the bones or treating them in a special apparatus with benzine. Steaming is effected in closed iron cylinders for a few hours at a pressure of 2 to 4 atmospheres.

The liquor thus obtained, which contains glue, fat and impurities, is drawn off and allowed to stand, whereby the fat separates on the surface. When cold the fat is skimmed off and remelted in water with the addition of common salt.

By extraction with benzine the bones are more completely exhausted than by steaming, but a strong odor of benzine, which can only be removed with difficulty, adheres to the fat. The latter is purified by remelting upon salt water and introducing steam for some time.

The fat obtained by boiling fresh bones has a white to yellowish color, a slight odor and taste, and is of a smearable consistency. It consists of stearin, palmitin and olein. When thoroughly purified it keeps well and makes a good lubricant. The bone-fat obtained as a by-product in the manufacture of bone-meal is largely obtained from old, partly putrefied bones, and is of a dark color and unpleasant odor. It always contains a considerable quantity of free fatty acids, and frequently calcium lactate and calcium oleate, the latter permitting the incorporation of considerable water.

The melting-point of commercial bone-fat ranges from 68° to 83° F. The fatty acids of the fat from fresh bones melt, according to Hübl, at 86° F. and congeal at 82.5° F. Valenta determined the saponification number at 190.9, and Demski and Morawski the iodine number of the separated fatty acids at 55.7 to 57.3. In examining bone-fat, attention must be especially directed towards dirt and water.

The ordinary bone-fat of commerce can only be bleached with difficulty, and frequently not at all, the methods with potassium dichromate and sulphuric or hydrochloric acid recommended for the purpose accomplishing the object in but few cases. The higher the percentage of free fatty acids, the more difficulty there will be in bleaching. According to Lewkowitsch products containing more than 50 per cent. of free fatty acids cannot be bleached. Fat obtained by extraction cannot be successfully bleached since, even when an apparently good result has been obtained, the dark color, as well as the disagreeable odor, soon reappears after bleaching.

As commercial bone-fats contain a large quantity of free fatty acids, they combine readily with strong lyes containing a considerable percentage of alkaline carbonates. In other respects their constitution varies, however, very much. There are, for instance, bone fats which in color and consistency resemble a poor quality of tallow and furnish a very good material for the manufacture of soap, while others resemble a poor grade of distilled oleic acid, and are incapable of forming cohering grain-flakes when boiled by themselves. Even with the better qualities of bone-fat, the spent lye is generally more or less turbid, and, on cooling, forms a gluey skin in consequence of the impurities contained in the fat. The yield varies very much. Good, solid bone-fat may yield as much as 150 to 155 per cent. of good ground grained soap, which is quite solid and lardaceous, though not so white as tallow grained soap.

Bone-fat by itself is but little employed in the manufacture of soap, but much in connection with other fats. It is chiefly used for rosined grained soaps, it being less adapted for smooth white grained soaps, as even the best bone-fat does not give the pure white product so desirable for this variety. It can also be used in connection with other fats for soft soaps, the lighter varieties being well adapted for figged soaps, however, only when the appearance of the product is of secondary importance, as is the case with soaps for the textile industry. For oil soaps only a small quantity can be worked in summer in connection with linseed-oil.

*Wool fat.*—The wool of sheep contains a considerable quantity of fatty substance which, however, can only be saponified with difficulty. Its chemical composition is not yet exactly known, but it is certain that it consists of a mixture of esters and free alcohols, among the latter occurring considerable quantities of cholesterin and isocholesterin. Moreover, the so-called wool-sweat (suint) contains considerable quantities of potash soap.

For the manufacture of soap, wool-fat is of but little value; it is chiefly used in the preparation of rosined soaps.

Wool fat possesses the property of forming with water emulsions which are readily absorbed by the skin, and various methods for its purification are known. In an anhydrous state the purified wool-fat is known as *adeps lanæ*, while the term *lanolin* has been applied to the product containing water; it serves as a vehicle for salves and pomades.

*Train and fish oils* are derived from various marine animals and are generally designated by the names of their sources, as whale, seal, cod-liver oil, etc. They are fluid at the ordinary temperature, and have a peculiar disagreeable odor and taste, due to the admixture of volatile fatty acids. The fatty acids occurring in the form of triglycerides in the train oils are but little known. In these oils, the oleic acid occurring in other oils, is partly replaced by physetoleic acid. The oils derived from several marine animals contain combinations which are not glycerides but ethers of the higher fatty alcohols.

All train-oils are sparingly soluble in cold alcohol, somewhat more freely in hot alcohol, and very readily in ether. Most of them are blackened by gaseous chlorine.

Train-oils are chiefly adulterated by compounding a good quality with a poorer one. Such sophistication can scarcely be detected, as it is difficult to distinguish the separate oils, their specific gravities varying but little, being between 0.915 and 0.930. Neither do the melting-points of the separated fatty acids, the saponification numbers and iodine numbers furnish a sufficient guide for the examination of train-oils.

An admixture of foreign fats is recognized by mixing 1 part of train-oil with 2 parts of concentrated sulphuric acid in a tall beaker. If the train-oil is free from foreign fat a clear mixture is obtained.

By cooling train-oils below 32° F. a solid fat is separated, which is known as *fish-tallow*. There is considerable difference in the commercial varieties of this product. Some of them have quite a pale color, a not disagreeable odor, and are of the consistency of tallow, while others show a very dark color, are smeary, and possess the true odor of train-oil. Fish-tallow

may be used for grained and household soaps. It saponifies readily with a 12-degree lye. If the clear paste free from scum is further treated with stronger lye it shows considerable firmness, but becomes tolerably light only upon the second or third water. The yield from a good quality of fish-tallow is up to 130 per cent., but much smaller from inferior qualities on account of their content of dirt and water. Even the best quality of fish-tallow without disagreeable odor shows in boiling the odor of train-oil, which can be noticed in the finished soap. It is therefore advisable to use the fat for soaps in the manufacture of which rosin is employed.

Train-oils formerly constituted the chief material, especially in Europe, for the manufacture of soft soaps, but their use for this purpose has been almost entirely abandoned, as the present demand is for soft soaps of as light a color and as slight an odor as possible.

## II. FATS AND OILS OF VEGETABLE ORIGIN.

The vegetable fats and oils applied to the manufacture of soap are very numerous and valuable, and are found in the fruits, seeds, etc., of plants. The most important solid fats are cocoanut oil, palm oil and palm-kernel oil. There are various other solid fats found in tropical countries, such as shea butter, illipe oil or bassia oil, piney tallow, Chinese tallow, cocoa butter, dika oil, butter of nutmeg, etc., which are very suitable for the manufacture of soap, but are little used on account of their high price, and will therefore be only briefly mentioned. Of the fluid vegetable fats are used: Olive oil, sesame oil, peanut oil, castor oil, cotton-seed oil, linseed oil, hemp oil, niger oil, cameline or German sesame oil, and small quantities of colza oil and poppy-seed oil.

*Cocoanut-oil.*—This valuable oil is obtained by boiling or pressing the ground or crushed kernels of the nuts of *Cocos nucifera*, the cocoanut palm, which is found in nearly all tropical countries. Most of the oil is imported from Ceylon and Cochin China, though recently the dried kernels, called

"coprah," are exported to Europe, where the oil is extracted either by expression or extraction. Coprah contains 60 to 70 per cent. of fat. Two varieties are known in commerce, sun-dried and kiln-dried coprah.

Fresh cocoanut-oil has a beautiful white color, a mild taste, and a peculiar, though not disagreeable, odor. It becomes soon rancid and acquires a somewhat disagreeable, acrid taste and odor. Entirely fresh oil melts at 68° F.; the commercial oil from Ceylon and Cochin China shows a melting-point of about 75° F., and the Brazilian cocoanut-oil (from *Elaeis bulyracea*) melts at about 80.5° F. By subjecting the coprah to a moderate cold pressure an oil is obtained which melts below 68° F. and congeals at 53.5° to 55.5° F., becoming heated thereby to 59° F.

Cocoanut-oil contains chiefly the glycerides of laurostearic, myristic, palmitic, and caprylic acids, and small quantities of capronic and capric acids. St. Evre and Bromeis claimed to have found special fatty acids, which they termed cokinic acid; but Heintz has shown that St. Evre's cokinic acid is a mixture of laurostearic acid with capric acid, and Bromeis's cokinic acid a mixture of laurostearic acid with myristic acid.

Cocoanut-oil has the highest saponification number of all the fats hitherto examined, by which it can be readily distinguished from all other fats, with the exception of palm-kernel oil, which comes next in this respect. Valenta examined several cocoanut-oils and found their saponification numbers to be from 257.3 to 268.4. The iodine number of cocoanut-oil is, according to Hübl, 8.9, and that of the separated fatty acids, according to Demski and Morawski, 8.39 to 8.79. The separated fatty acids melt at 76° to 77° F., and congeal at 66° to 69° F.

On account of its large percentage of laurostearin, the action of cocoanut-oil in the process of saponification is quite different from that of tallow and most other fats. It requires strong caustic lyes and forms soaps which can only be separated by concentrated brine, and then become so extraordi-

arilly hard that they cannot be cut. For this reason a clear boiling to the solid would in case of cocoanut-oil soap be entirely contrary to the end in view and very difficult. While, furthermore, tallow, for instance, treated with very strong lye, floats on top and then can scarcely or not at all be saponified, in the case of cocoanut-oil just the contrary happens. It does not form that milk-like mixture (emulsion) with weak lyes by which the process of saponification is usually preceded, but floats as a clear fat above; only when, by continued boiling and evaporation, the lye has reached a certain strength, the saponification suddenly ensues. For saponifying cocoanut-oil lyes of such strength are used that the soap, with the lye, receives the intended contents of water, and a separation becomes, therefore, unnecessary. Of course, the amount of the alkali must be so accurately calculated that the soap receives no excess of alkali, or at least but very little.

Cocoanut-oil can also be saponified in the so-called cold way, *i. e.*, can be converted into soap by stirring in strong caustic lyes at a temperature little above the melting-point of the oil. Soaps from cocoanut-oil have the property of absorbing a great deal of water without suffering in firmness and appearance. They dissolve freely in water, yielding a heavy lather, which is, however, not so durable as that from tallow soap. Soaps from pure cocoanut-oil have the disagreeable property, even if they do not contain an excess of alkali, of producing a burning sensation and redness upon a sensitive skin, and being strongly inclined towards rancidity, whereby they acquire a bad odor and unsightly appearance.

Three principal kinds of cocoanut oil are at present known in commerce—Cochin China oil, Ceylon oil and coprah oil—the first being much the best and *purest* in color. It is best adapted for cold saponification, but is only good when not too old. When old oil of a certain degree of rancidity is stirred together with strong lye, the mass becomes thick too quickly and the soap shows formation of granulation. Commercial Ceylon oil is generally quite rancid, and therefore not well

adapted for cold saponification, and besides, the soaps prepared from it are not pure white, but of a grayish shade. Coprah oil is not very rancid, and therefore suitable for cold saponification; it does not yield, however, pure white soaps, and hence is not suitable for toilet soaps. This defect can, however, be remedied by the following method of refining: Boil 750 pounds of coprah oil with 15 pounds of soda lye of 6° B. and 10 pounds of water for half an hour, constantly removing the scum. Then add 1½ pounds of common salt, remove the scum, and let the whole boil half an hour longer. Repeat the boiling with 1½ pounds more of common salt and let the oil stand over night for the salt water to settle.

*Palm oil.*—Vast quantities of this oil are consumed in the manufacture of soap, especially in England, where it was first used. It is obtained by boiling or pressing the fruits of various species of palms, chiefly of *Aroira elaeis* or *Elaeis guianensis* and *Elaeis melanococca*. The principal places of production are Western Africa (Guinea) and South America (Guyana), while some of it is brought into commerce from the Canaries, Madeira, and other places. The largest quantity and best quality of palm oil come from the West African coast south of Sinoe, in the Republic of Liberia, to Cameroon, in the Bay of Benin. The fruits of *Aroira elaeis* are dark orange-yellow, almost brown, of the size of a pigeon's egg, and contain a solid kernel under a fleshy cover. The latter yields the product known as palm oil, which is directly brought into commerce from the localities where the palms grow. The kernels also yield an excellent oil, known as palm-kernel oil, the manufacture of which is, however, principally carried on in Europe, where it is either expressed or extracted by means of carbon disulphide or benzol.

Palm oil is of an orange-yellow to red-brown color, of a butyraceous consistency, and, when not rancid, has the odor of orris-root. The melting-point of fresh palm oil is at about 80.5° F., while that of rancid oil is much higher, rising to 108.5° F. The melting-point of the fatty acids separated

from palm oil ranges between 116.5° and 118.5° F., and their congealing point between 104° and 113° F. The saponification number of palm oil is, according to Valenta, 202 to 202.5, and that of the separated fatty acids 206.5 to 207.3. The iodine number is, according to Hübl, 51.5.

Palm oil consists chiefly of palmitin and olein. A very characteristic feature of this fat is the great quantity of free fatty acids it contains. In fresh palm oil has been found one-third and in an old sample as much as four-fifths of its weight of free fatty acids. The greater portion of glycerin separates as such, and can be obtained by extraction with water.

The reddish-yellow color of palm-oil is not destroyed by saponification, so that the soap manufactured from the crude product shows a yellow color. In the manufacture of stearin the coloring substance can be destroyed by acid saponification and distillation, but not by autoclave and ordinary lime saponification. Bleaching the oil may be effected by superheating, by heat and air, and by chemical agents. However not all varieties of palm-oil can be bleached. Lagos and Old Calabar oils give good results, but dirty red oils, such as Congo oil, cannot be bleached.

*Superheated steam i. e.* rapidly heating the oil to between 428° and 464° F., is the simplest method of bleaching. Impure oil must, however, first be purified by melting upon water at a moderate heat. The supernatant clear fluid is then drawn off from the sediment, brought into an iron kettle, and quickly heated to the above mentioned temperature, one of 428° F. sufficing for most varieties, though some require a higher one.

A method of bleaching by superheating recommended by Klepzig is as follows: The oil is quickly heated to 392° F. and then carefully to between 419° and 428° F., the latter temperature being kept up for one hour, without stirring. In about half an hour the oil has acquired a lemon color and is perfectly clear, the yellow coloration disappearing, as a rule,

entirely in  $1\frac{1}{2}$  hours, and the oil becoming dirty gray. By pouring a few drops of the oil upon a plate, fine particles of carbon will be observed floating in them. The fire is then withdrawn and the oil allowed to stand quietly in the kettle. With some experience it can be readily judged whether the oil is to be kept for some time longer at the indicated temperature, or heated to a still higher degree.

Oil purified before heating shows, when cold, a whitish color shading into brown; oil not previously purified is dirty gray from the many finely-divided particles of carbon suspended in it. These impurities, however, do not injure the oil for the manufacture of soap, as they are removed by salting out. Klepzig is of the opinion that 'bleaching' is effected by this finely-divided carbon; this, however, is not correct, since well-purified oils, in which the formation of carbon is very slight, are also bleached by the application of heat. This method of bleaching simply depends upon the fact that the coloring matter of palm-oil is destroyed by heat. It has the defect that the bottoms of the kettles suffer much. With careful work and good, pure palm-oil the loss in weight is 1 to  $1\frac{1}{2}$  per cent.

To bleach palm-oil by the *agency of heat and air*, it is heated to  $212^{\circ}$  F., and at this temperature brought in contact with the air which is accomplished by various mechanical appliances. The most simple is to fill a fine-meshed sieve fastened to a long handle with oil, raise it up quickly, and bring the oil in contact with the atmospheric air by allowing it to run back into the kettle. By frequently repeating this rather slow process the oil is bleached. A better arrangement is a sort of paddle wheel so adjusted that one-half of it dips into the oil. By setting the wheel in motion the paddles lift up a portion of the oil, allowing it to run back in a fine stream into the kettle. This mode of bleaching has the advantage of not impairing the agreeable odor of the oil. It is, however, not available for inferior qualities of oil which can at the best be only bleached by chemical agents. From the color of the oil

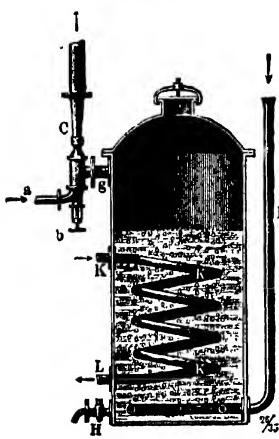
a conclusion can readily be drawn as to its bleaching capacity. The more the color passes from reddish into brown, the more difficult it will be to bleach. Soaps from palm-oil bleached by light and air turn white far more slowly than soaps from chemically bleached oils.

Koerting's steam-jet air-suction apparatus Fig. 5 is very

suitable for bleaching with heat and air. *C* is the air-suction apparatus; the steam enters at *a*,

and is regulated by *b*; *g* is the sucking-off pipe; *K* a steam heating coil; *H* the stop-cock for discharging the oil and *E* the pipe for conveying the air.

The apparatus works as follows: The oil in the kettle is first heated by means of the steam-coil to about 212° F. The air is then sucked in and produces a vacuum above the oil. The air passing in through the air-pipe, as well as the vapors evolved from the oil, are sucked off below the vacuum.



off below the vacuum. The atmospheric air introduced through the air-pipe passes out through a large number of small holes near the bottom of the kettle and, while ascending is intimately mixed with the oil. In this manner 20 to 25 hundredweight of palm-oil can in about 2 hours be nicely bleached. The end of the bleaching process is recognized by taking samples through the cock *H* and allowing them to cool upon a porcelain-dish. This process of bleaching gives excellent results if the oil can be bleached by the oxygen of the air alone. It is, however, absolutely necessary for the oil to be free from dirt and water, as otherwise the bleaching operation will not be successful. To obtain crude oil of sufficient purity it has to be melted the day before, and allowed to settle overnight.

*Bleaching by chemical agents* is effected by oxidizing substances, the best being chromic acid, *i. e.*, a mixture of potassium dichromate and hydrochloric acid. The process as executed in most soap factories is as follows: The palm-oil is first purified by melting upon water. After allowing the impurities to settle the supernatant clear oil is drawn off and allowed to cool to 122° F. To 1000 pounds of oil are then added, with constant stirring, 50 pounds of hydrochloric acid and 12 pounds of potassium dichromate previously dissolved in 24 pounds of boiling water. After stirring ten to fifteen minutes the oil shows a dark, dirty gray color. Sometimes a few pounds of sulphuric acid are added. The stirring is continued until the oil is entirely clear with a bluish luster. 60 to 80 pounds of hot water are then poured over the oil by means of a sprinkler, and after covering the vat it is allowed to stand till the next day.

With most varieties a much smaller quantity of potassium dichromate suffices, 1000 pounds of oil frequently requiring only 5 to 6 pounds.

A process of bleaching palm-oil, recommended by Bolley, is as follows: 1000 pounds of palm-oil, previously melted, are brought at 122° F. into a wooden vat; 5 pounds of potassium dichromate are then added, and, after thorough stirring, 10 pounds of hydrochloric acid, and finally  $\frac{1}{2}$  pound of sulphuric acid. Stirring is then continued half an hour and a sample taken. If the oil proves still yellow, some more potassium dichromate, hydrochloric acid and sulphuric acid are added until the desired result is obtained. The oil is then brought into a copper kettle and boiled for some time with water to remove all constituents of the bleaching agent. It is then allowed to stand covered for some time, and finally the supernatant clear fat drawn off.

Palm-oil bleached by means of potassium dichromate has frequently a greenish shade, which is due to a small quantity of chromic oxide contained in it. This can be extracted, and a faultlessly white product obtained by boiling with

dilute hydrochloric acid and remelting the oil upon pure water.

Commercial palm-oil varies very much in quality, the *prima lagos* and *secunda lagos* being the best, the former being more readily bleached than the latter. Next in quality is the *old Calabar oil*, which is also readily bleached, especially by chemical means. The oils from *Accra* and *Benin* are not so pure as the preceding ones, and very unequal in their behavior during bleaching. They are generally used for dark grained soaps and rosined grained soaps. The crude oils from *Cameroon*, *Gaboon* and *Liberia* are generally remelted on board the vessels, and brought into commerce under the name of *purified Cameroons*. They are generally very rancid, and, notwithstanding their name, by no means free from dirt. They cannot be as readily bleached as lagos and old Calabar oil, and generally require chemical agents for the purpose.

To obtain the oil from the sediment formed by storing large quantities of palm-oil, the sediment is boiled upon water, and after standing for some time the supernatant clear oil drawn off. The slimy residue is brought into large barrels, and after adding, with constant stirring, some sulphuric acid, and continuing the stirring for some time, the barrels are covered and the contents allowed to settle. After a few hours the dark-colored oil, which has separated, is drawn off, and can be either bleached with potassium dichromate and acid, or immediately used for dark soaps.

The sediment obtained by bleaching with chemical agents also contains much oil, and is therefore washed with hot water with an addition of sulphuric acid. The oil obtained, though dark, yields quite pale soaps, which in odor and firmness are not inferior to other palm oil soaps.

Palm oil was formerly much adulterated, and substitutes for it consisting of mixtures of wax, tallow, and lard colored with turmeric and scented with orris-root, were even found in commerce. Such adulterations and mixtures could be readily detected by the test with glacial acetic acid, which dissolves

palm oil, but leaves all the rest undissolved. The addition of turmeric is recognized by the brown color formed on stirring in soda lye. With the present prices of palm oil such adulterations need, however, not be feared, as they would not pay.

All palm oils, no matter how much they may vary in quality, yield a firm soap of an agreeable odor, the scent remaining quite perceptible in combination with other fats and oils, and even with rosin.

Palm oil, crude or bleached, is readily saponified. It yields, even with weak lye of 8° B., a quite thick and viscid soap paste. Generally it is, however, saponified with lye of 12° to 15° B., yielding, when thoroughly salted out, an abundant grain quite free from scum, which, if the paste was entirely clear, is thoroughly saturated. Since palm oil is generally quite rancid, and contains, therefore, free fatty acids, a lye of 15° B., which, with a high percentage of lime, always contains some alkaline carbonate, is completely exhausted, especially when adding it carefully towards the end of the operation and boiling slowly. Saponification with direct steam is especially quick and perfect, the grain separating almost entirely clear, the best proof of a complete saponification.

By reason of the large content of palmitic acid, palm oil yields a good firm soap, notwithstanding the large percentage of water retained even by grained soap. 100 lbs. of pure oil yield 162 to 165 lbs. of fresh ground grained soap.

The principal use of palm oil, besides the manufacture of soap, is in the preparation of stearin.

*Palm-kernel oil*, usually called *kernel oil* by the soap boiler, is obtained either by expression, or by extraction with carbon disulphide or benzine from the kernels of the palm-fruit. The kernels reach the market freed from the woody shell. The percentage of oil varies between 35 and 50 per cent., though some kernels, it is claimed, contain as much as 60 per cent. and more.

Palm-kernel oil consists chiefly of the glycerides of lauric, stearic, palmitic, and oleic acids, and contains only small

quantities of tricaprin, tricaprylin, and tricapronin (and perhaps some trimyristin) in about the following proportions: Triolein, 26.6 per cent.; tristearin, tripalmitin (trimyristin?) 33 per cent.; trilaurin, tricaprin, tricaprylin, tricapronin 40.4 per cent.

The melting-point of fresh palm-kernel oil is at from 77° to 79° F.; that of old rancid oil being somewhat higher. Its saponification number is, according to Valenta, 247.6, and that of the separated fatty acids 265.8. By the determination of the saponification number the fatty acids of palm oil can be readily distinguished from those of palm-kernel oil. The iodine number of palm-kernel oil is, according to Hübl, 13.4 to 13.6, and that of the separated fatty acids, according to Demski and Morawski, 12.07.

The behavior of palm-kernel oil, as regards saponification, resembles that of cocoanut oil; the behavior of both these fats, though not exactly alike, being, no doubt, due to the large percentage of laurostearin characteristic of them. Palm-kernel oil requires strong caustic lyes, though of a less degree than cocoanut oil. While palm-kernel oil combines quickest with an initial lye of 26° to 30° B., and the combination takes place with greater difficulty the more these degrees are exceeded, cocoanut oil readily forms a combination with much stronger lye. In their behavior towards common salt the soaps from the two fats also resemble each other, but are not alike. Soaps from palm-kernel oil are salted out with difficulty though with greater ease than soaps from cocoanut oil. While with the latter soaps an augmentation of up to 1200 per cent. yield by salt water is possible, the resulting product being firm, with soaps from palm-kernel oil an augmentation up to from 600 to 700 per cent. is at the utmost possible.

It has been frequently asked whether there is a difference between expressed and extracted palm-kernel oil; at the present time this question can be unreservedly answered with "No." Cases, formerly referred to by different writers, of soap from extracted oil turning entirely black in boiling, with

the development of an intense odor of carbon disulphide, are scarcely possible now, and were, no doubt, due to extracting the oil with impure carbon disulphide and insufficient subsequent purification.

It has recently been observed that palm-kernel oil extracted with carbon disulphide turns green in saponifying. The green color, however, disappears again after the lye required for complete saponification has been added.

The manner of detecting carbon disulphide in extracted oil has previously been given. The oil can be freed from carbon disulphide, or rather from its products of decomposition, by boiling it for some time with salt water by the introduction of steam. To test whether the desired result is attained, take from time to time a sample of the oil, filter it through paper until entirely clear and saponify it. The resulting soap must not show a bad odor.

*Shea butter, galam butter or bambuk butter* is obtained by boiling with water the comminuted kernels of *Bassia parkii*, a tree belonging to the *Sapotaceæ* species, growing in the interior of Africa. The fat is of a butyraceous consistency, gray-white or greenish-white, of a peculiarly viscid and sticky nature (similar to a mixture of fat and turpentine), and a peculiar aromatic odor. It keeps a long time without turning rancid. It melts at between 82.5° and 84° F., though statements of various observers vary very much in this respect.

For the manufacture of soap, shea butter can only be used as an addition to other fats, it being best employed in connection with palm-kernel oil.

For rosined grained soaps, with an addition of rosin of up to 25 per cent., shea butter together with palm-kernel oil is very suitable, the result being a very fair product. However, for soaps which are subsequently to be filled, shea butter has to be used with great precaution.

On account of its great content of non-saponifiable substances, shea butter gives only a yield of 130 per cent.

*Illoopa oil or Bassia oil*, also called *Mahwa butter*, is obtained

from the seeds of *Bassia longifolia* and *Bassia latifolia*. The fresh fat is yellow and has a bitter aromatic taste and a characteristic odor reminding one of cacao beans. On exposure to air and light the coloration disappears quite rapidly and the fat becomes rancid.

According to Dirks, the use of Bassia oil for the manufacture of soap has been very unsatisfactory. Though the soaps are not as brittle as those prepared from shea butter, they cannot be called faultless as regards firmness and durability. Bassia oil to be used for white grained soaps has to be previously boiled, as it contains considerable quantities of mucilage and other impurities. In working larger quantities of the oil for grained soaps, the separation of vegetable impurities may be so great that a dark green, viscous, non-saponifiable coat is formed on the sides and bottom of the kettle. This coat, when cold, is so viscous that it can only be removed with difficulty from the sides of the kettle. In buying Bassia oil, 15 per cent. must be allowed for impurities, and, to use the oil to advantage for saponification, it must be 20 per cent. cheaper than palm-kernel oil. For grained soaps, which are subsequently to be filled, it has to be used with caution, as it brings too many foreign substances into the soap which effect the stability of the combination.

*Piney tallow, also called vegetable tallow or vateria fat*, is obtained from the seeds of *Vateria indica*, L., a tree indigenous to the East Indies. The air-dried seeds contain 49.21 per cent. of a solid, greenish-yellow fat, which soon becomes white on exposure to light, and has a peculiar slightly balsamic odor. The fat prepared from commercial seeds shows a melting-point of 107.5° F., while, according to other statements, it melts at 98° F. and congeals at 87° F. It saponifies with great ease, 1 grammie of fat requiring 191.9 milligrammes of caustic potash, whereby 8.4 per cent. of glycerin is separated. The fatty acids separated from the products of saponification melt at 134° F. and congeal at 130.5° F., and consist of a mixture of oleic acid and solid fatty acids. The latter amount

to about 60 per cent. of the weight of the vegetable tallow, and melt at 147° F. Notwithstanding this high melting point, the pre-eminently crystalline product is rather soft and readily friable.

According to Derks, piney tallow boiled by itself saponifies with a lye of 12° B. to a clear paste, the result in salting-out being a fine closed grain with a yield of about 142 per cent. The fat is very suitable for all kinds of rosined grained soaps. Although the pale soaps acquire at first somewhat of the green coloration of the fat, they soon bleach satisfactorily.

*Chinese tallow*, also called *vegetable tallow* and *Stillingia tallow* is obtained from the seeds of *Stillingia sebifera*, or the tallow tree indigenous to China. The fruit of the tree contains three egg-shaped seeds coated with a thick hard tallowy layer, while the seed kernel contains a liquid fat. The tallow is obtained by one of two methods. The seeds are either freed from the capsules and treated with steam whereby the tallow—about 20 to 30 per cent. of the weight of the seeds—melts off; the seeds are then crushed to obtain the liquid fat. Or, the seeds are at once crushed and the tallow and liquid fat are boiled out together with water. The tallow obtained by the first method is white or greenish-white, inodorous, quite hard, and melts at 104° to 105° F.; it consists chiefly of palmitin. The fat obtained according to the other method forms a whitish or gray-white mass with a slight odor; it becomes yellow to brown by storing, shows a slightly acid reaction and congeals at about 95° F.

Chinese tallow is said to have for some time been used in England in the manufacture of candles and soap. As regards its hardness and yield it can, according to L. Borchert, be classed with the best varieties of tallow. In boiling it behaves like the best quality of animal fat, and yields very hard soaps which, as to detergent power and economy in use, remind one of stearin soaps. As Chinese tallow, by reason of the primitive methods of its production, contains considerable dirt, Dirks recommends to purify it before use. For the reason mentioned, the yield amounts only to about 142 per cent.

Commercial Chinese tallow varies very much in quality. The melting-point of the neutral fat, which is always much below that of the fatty acids, differs considerably, being sometimes as low as 95° F. The fatty acids generally drain off at 131° F.; with pale yellow tallow the temperature, however, rises to nearly 140° F., and with green tallow sinks to 120° F.

Chinese tallow contains frequently considerable water, though in examining it special attention should be directed towards dirt in the canisters, of which there is frequently found an astonishing quantity.

Under the name of "vegetable tallow" another vegetable fat is brought into market which is obtained, chiefly in Borneo, from various species of *Hopea*, especially *Hopea splendida* and *Hopea aspera*. The ripe fruits are piled up in a moist place and allowed to germinate a little. They are then dried in the sun until they are brittle, when they are deprived of their shell and put into a rattan or bamboo basket suspended over boiling water. The arising steam renders the fruits soft and plastic. The fat is then expressed by squeezing the doughy mass in a cloth, and is poured into joints of bamboo, by which it receives the cylindrical form in which it is met with in commerce. By the natives the tallow is used for culinary and lighting purposes. It is largely exported to England, where it has been successfully employed as a lubricating agent, being very valuable for this purpose, far surpassing even olive oil. In Manila it is employed in the manufacture of candles.

The tallow is white and hard and crumbly at an ordinary temperature, but softens at a moderate heat, and has a somewhat nutty taste and odor. According to Fielding, it remains hard up to a temperature of 64.5° F., softens between 80.5° and 104° F. to a pasty mass, and melts at 112° F. It dissolves in about half its weight of cold ether, is sparingly soluble in cold, but freely in hot, acetone; on cooling the greater portion is, however, reseparated. It dissolves in half its weight of chloroform, is readily soluble in oil of turpentine, and very freely in carbon disulphide, and in hot benzene. It is also soluble in about 30 parts of cold and 20 parts of hot alcohol.

*Cocoa butter* is obtained as a by-product in chocolate factories by hot expression of the cocoa bean, the fruit of *Theobroma cacao*, L. In a fresh and pure state the fat is yellowish, but becomes nearly white with increasing age. It smells like roasted cocoa beans and keeps a long time without becoming rancid, and for this reason is much employed for cosmetic and pharmaceutical preparations. The specific gravity of fresh cocoa butter is 0.950 to 0.960, and that of old 0.947 to 0.950. The statements in regard to the melting-point vary between 77° and 91.5° F., the variations being, no doubt, chiefly due to the methods employed. Melted cocoa butter can, according to Rüdorff, be readily cooled to 71.5° F., and on congealing shows a permanent increase of temperature to 82° F. Cocoa butter gives a clear solution with ether, oil of turpentine, and chloroform. Its saponification number is 198 to 203, and its iodine number 51.0. The fatty acids separated from it melt at 125° F. and congeal at 123.5° F.

The quality of cocoa-butter may be partly determined by the odor, taste and consistency. It is frequently adulterated especially with palm-kernel oil, cocoa-stearin, as well as with dika oil and beef tallow. For the detection of the first two adulterants the determination of the saponification number and iodine number is generally sufficient, the former being considerably higher in their presence, and the latter considerably lower than with the genuine product.

For the detection of tallow Bjoerklund's ether test is recommended: Pour 6 grammes of ether over 3 grammes cocoa-butter, close the test-tube with a cork, and effect solution by shaking at 64.4° F.; the sample should form a clear solution. Then place the test-tube in water of 32° F. If the previously clear solution becomes turbid in 10 to 15 minutes and again clear at 66.2° to 68° F., the fat is pure. If the fat is adulterated with beef-tallow turbidity appears sooner—in 7 to 8 minutes—and the solution again clarifies only at 71.6° to 77° F.

No sure means of detecting the presence of dika oil has thus far been found.

*Dika oil, or wild mango oil*, is obtained from the seeds of *Mangifera gabonensis*, a tree indigenous to the west coast of Africa. When fresh it is pure white and of a mild cocoa-like odor and taste, but when stored for some time it becomes yellow and rancid. It melts at from 86° to 89.5° F., contains the glycerides of lauric and myristic acids, and is readily saponified.

*Olive oil* is obtained from the fruit of several varieties of the *Olea Europaea*, the quality of the oil varying slightly with the class of tree yielding the fruit. The nearly ripe olives contain 30 to 50 per cent. of oil and are generally first submitted to slight pressure, whereby the *virgin oil* is obtained. The yield of this is very small, the bulk of the oil being obtained by crushing the flesh in edge-runners or between rollers, followed by moderate pressure, the first product being the best. The *marc* or *press-cake* is broken up, stirred with boiling water, and then more strongly pressed; the second marc is similarly treated, the stones also being crushed, and very strongly pressed. Each succeeding extraction yields an oil inferior to that preceding it; and the residual oil in the final press cake may be obtained by extraction with volatile solvents. Such press-cakes are known in Italy as *Sanza*, and carbon disulphide is as a rule used for extracting the oil still contained in them. This oil is generally known as *sulphur oil*.

Inferior olive oil, not fit for dietetic purposes, but used for lubrication, although containing 7 to 8 per cent. of free oleic acid, is known as *gallipoli* or *engine oil*. Sometimes the kernels are crushed or extracted separately, yielding *olive-kernel oil*, which is similar to low-grade olive oil.

Olive oil possesses the color characteristic of the fruit, the green tinge being due to chlorophyll. It has a mild flavor when fresh, but becomes rancid more readily than true seed oils. It dissolves sparingly in alcohol, but readily in 1½ to 2½ parts ether, and in 3 parts acetic ether. Cold-pressed oil consists of about 70 per cent. of glyceryl salts (chiefly oleate) liquid at the ordinary temperature, the remaining 30 per

cent. being chiefly glyceryl salts of fatty acids of the acetic series (notably palmitic acid); these partially separate on cooling to about 50° F. The hot-pressed oils are richer in palmitin. The specific gravity of cold-pressed oil ranges at 59° F. between 0.915 and 0.918, while that of hot-pressed oils is frequently as much as 0.925. The latter deposit granular secretions at 50° F. and congeal at 32° F., while superior grades of cold-pressed oil become only turbid at 35.5° F., and separate palmitin at 21° F. The fatty acids separated from olive oil melt at from 71.5° to 79° F., and congeal at from 70° to 71.5° F. The saponification number of olive oil is 191 to 192, and the iodine number 81.6 to 84.6, and that of the separated fatty acids 86.1. The better grades of olive oil are frequently adulterated, particularly with sesame oil and cotton-seed oil. An excellent means of recognizing pure olive oil is the iodine number, as nearly all oils used for adulteration show a higher number.

Olive-oil foots and the oil obtained by extracting the press-cakes with carbon disulphide, which is generally known as sulphur oil, are chiefly used in the manufacture of soap, they alone yielding hard bar-soaps, while the liquid oils without an addition of cocoa-nut oil or other solid fats can only be employed for soft soaps. Like tallow soaps, olive-oil soaps stand but little salt, and can be salted out with 5 to 8 per cent. of the latter. There is however considerable difference as regards saponification between olive-oil foots and sulphur oil. While the former requires a weak lye for the induction of saponification, the latter saponifies with any kind of lye. Sulphur oil is generally saponified with caustic soda lye of about 25° B. The soap is then separated from the brine and sufficiently ground for the dirt and coloring matters to separate. The finished soap has a thin, dirty green cover of froth. Beneath this lies a bright olive-green grain and between the latter and the thick sub-lye a highly colored soap which is worked either by itself or added to the next boiling. The grained soap is moderately solid, somewhat like soap from half

tallow and half lard. Soap from oil which has been extracted from fresh residues has not a disagreeable odor, but, if previous to extraction, the press-cake or sanza has passed into fermentation, a lasting offensive odor adheres to the oil as well as to the soap made from it.

From 100 lbs. of sulphur oil, 150 to 160 lbs. of ground or fitted grained soap are obtained; however under certain conditions the soap is capable of absorbing much water as well as silicate of soda and talc.

*Sesamé oil* is obtained by crushing the seeds of *Sesamum orientale*, commonly called *til-seed*. The seeds contain 47 to 56 per cent. of oil. The latter has a pale yellow color and contains a small quantity of a resinous body which can be extracted by repeated shaking with glacial acetic acid. The oil has a specific gravity of 0.921 to 0.924. It consists chiefly of olein and in addition contains the glycerides of linoleic, stearic and palmitic acids. Its saponification number is, according to Valenta, 190, and that of the fatty acids separated from it, 199.3; the latter melt at from 77° to 79° F., and congeal at 72° F. The iodine number of the oil is from 102.7 to 106 and that of the fatty acids separated from it, 108.9 to 111.4.

With sugar and hydrochloric acid, sesamé oil gives a crimson color, and it can thus be detected in a mixture with other oils. This test is executed as follows: Dissolve in a test-tube 0.1 gramme cane-sugar in 16 cubic centimeters hydrochloric acid of 1.19 specific gravity, add 20 ccm. of the oil to be examined, shake vigorously for a short time and allow to stand; the aqueous solution separates in a very short time and shows a crimson color even in the presence of the smallest quantity of sesamé oil.

Sesamé oil is used as a food in place of olive oil, and is sometimes mixed with the latter as an adulterant, while in its turn it is frequently adulterated with peanut oil. For the manufacture of soap the oils obtained from the last pressure and those pressed or extracted from spoiled seeds are only used. For bar soap the thick, almost white, foots which

deposit in storing the oil, and contain much stearin or palmitin, can only be employed. The liquid oil may, however, be worked as an addition of up to 30 per cent. to palm-kernel oil, cocoanut oil, tallow and palm oil, and then yields good solid soaps.

Oil obtained from bad, mouldy seed has a very disagreeable odor, which, however, can to a great extent be removed by boiling the oil upon salt water with steam. It is then, while still hot, drawn off into large reservoirs and allowed to rest. On cooling, a large quantity of solid fat, almost white, is separated, which is very suitable for bar soaps.

Sesamé oils saponify readily, behaving in this respect similar to lard. In boiling with caustic soda lye, the foots are gradually added to the boiling lye, 200 pounds of the latter of 15° B. being as a rule allowed for 100 pounds of oil. Saponification of the oil takes place already when only about half of the latter in proportion to lye is in the kettle; but even when sufficient oil has been added to the lye a long, smooth-paste-soap is not obtained. Hence the correct proportion between fat and lye has to be previously calculated, and accordingly added and boiled till it has pressure. Salting out should not be effected so long as the soap is smeary, otherwise the latter remains so after salting out, and retains, even after grinding, lye and particles of dirt. After salting out, the soap boils bright, with a large grain, and is entirely free from froth. To harden it still more, salt-lye is withdrawn from it till it can no longer boil high. Samples of the soap taken at this stage are hard and, when cold, crumble, similar to moist flour.

Pure liquid sesamé oil saponifies similar to linseed oil and with difficulty with pure soda lye; it does not yield a good grained soap.

*Peanut oil or earth-nut oil* is obtained from the fruit of *Arachis hypogaea*, by drying and pressing, some 40 per cent. of oil being obtained as the gross yield of several treatments, while 45 per cent. is about the content of the seeds. The cold drawn oil is used for eating instead of olive oil, the remaining

portions serving for lighting and soap-making. The pressed oil-cake containing about 8 per cent. of oil is used as cattle feed.

Peanut oil is somewhat more limpid than olive oil, and contains the glycerides of oleic, linoleic, stearic, hypogaeic and arachidic, and perhaps of other, acids. The specific gravity of the finest quality of fresh oil is 0.916 at 59° F., while that of the second and third pressures is as much as 0.920. Peanut oil belongs to the non-drying oils; it is quite durable and does not very soon become rancid. It becomes turbid at 37.4° F., congeals at 26.5° F., and solidifies entirely at 19° F. Its saponification number is from 191.3 to 191.6; its iodine number, according to Hübl, 103, and, according to Moore, 87.3. The separated fatty acids melt at 82° F. and congeal at 75° F.; their iodine number is from 95.5 to 96.9.

Peanut oil is used in the manufacture of grained soap, half-boiled soap and soft soap. Lyes of not less than 18° B. are used for saponification. The oil in many respects resembles cotton-seed oil, but has the advantage that the soaps do not show yellow stains. Two parts of peanut oil and 3 parts of palm-kernel oil, boiled directly with caustic soda lyes, yield faultless wax-grained soaps. Beautiful half-boiled soaps are obtained from 70 per cent. of palm-kernel oil and 30 per cent. of peanut oil, with caustic soda lye. It is best to boil in the direct way, using pure caustic lye of about 24° B., and afterwards reducing causticity by solution of soda or common salt.

As regards soft soaps, peanut oil is especially adapted for the manufacture of so-called silver soap, or elaidin soap, being preferable for this purpose to cotton-seed oil. For cold-stirred soaps the use of peanut oil is of great advantage. A stock, for instance, of 70 pounds of cocoanut oil, 30 pounds of peanut oil and 60 pounds of caustic soda lye of 36° B. yields a product superior in appearance to a soap in which tallow is used instead of peanut oil. Peanut oil is also well adapted for the preparation of stock soaps for toilet soaps. For transparent glycerin soaps, peanut oil cannot, however, be substituted for tallow, as the soaps would become too soft.

*Castor oil* is obtained from the seeds of *Ricinus communis*, originally indigenous to the East Indies, but now cultivated in Algiers, Egypt, Greece, Italy and America. The crushed seed was formerly twice cold pressed, and the press cake was then cut up, moistened with hot water, heated in an oven and subjected to a third pressure. However, the general practice at the present time is to extract by one pressure, by means of a powerful press, the oil from the crushed seeds, so that only 5 to 6 per cent. of it remains, in most cases, in the press cake. It has been attempted to regain this oil by extraction, but the process offers no advantage, as the value of the press cake as a fertilizer is thereby impaired. Since the poisonous alkaloid *ricin*, which is contained in the seed, remains in the press cake, the latter cannot be used for feeding purposes.

The heavy, sound seed is selected for the preparation of castor oil for medicinal use; the oil, however, only becomes fit for the purpose by clarifying and filtering. Defective seed and the residue from the filtration of the first quality of oil are used for oil of second quality. Such oil is partly used in the manufacture of toilet soaps. Another grade of oil derived from the foots, residues, etc., is of a very dark color, and frequently possesses a very disagreeable odor. It is employed for lubricating and for making Turkey-red oil.

Castor oil is colorless or slightly yellowish, of a mild taste, with an acrid after-taste and a slight, though not disagreeable, odor. It is chiefly composed of the glyceride of ricinoleic acid and a small quantity of stearin and palmitin. Its specific gravity varies between 0.95 and 0.97 at 59° F. At 32° F. it deposits a white stearin-like fat, and at 1.4° to —0.4° F. congeals to a yellowish, transparent mass. American castor oil frequently separates stiff fat at 42.5° F., and congeals at from 14° to 10.5° F.

The saponification number of castor oil is, according to Valenta, from 181.0 to 181.5; its iodine number is 84.4, and that of the separated fatty acids 86.6 to 83.3. The fatty acids melt at 55.5° F., and congeal at 37.5° F.

The value of castor oil is determined by the color which should be as light as possible, and by the odor. Dark-colored oil has suffered from light or air, and badly smelling oil has been pressed from spoiled seed and residues. Pure castor oil does not become rancid even if kept for a long time.

The behavior of castor oil as regards saponification is similar to that of cocoanut oil, it being readily saponified by stirring together with strong soda lye. The resulting soap is very white, amorphous and transparent, and quite hard even with a content of as much as 70 per cent. It possesses also the property of dissolving in pure cold water without rendering the latter turbid or opalescent. Castor oil is chiefly used in the manufacture of transparent soaps.

*Cotton-seed oil* is obtained from the seeds of the cotton plants, *Gossypium herbaceum*, *G. arboreum*, *G. barbadense*, and other varieties. The seeds having been screened from all dust and foreign substances are freed from adhering cotton by passing them through a machine similar to a gin, only with teeth placed more closely together. They are then brought into the huller, which consists of a cylinder armed with steel blades, and surrounded about two-thirds way by a concave box also armed with corresponding knives. The cylinder revolves at great speed, and as the seeds are forced between the knives the pericarps or hulls are broken and forced from the kernels. The mass then falls into a large revolving sieve. The kernels, many of which are broken into fine pieces, pass through the meshes of the sieve and the hulls are carried away. The clean seeds are now crushed between rolls and the resulting meal heated in a pan until the water it contains is evaporated. The hot meal is then placed in wedge-shaped bags of woolen duck, each holding sufficient for a cake. The bags are placed between the sides of wrappers formed of woven horse-hair backed with corrugated leather to facilitate the escape of the oil, and subjected to pressure in a hydraulic press. Some modern presses are so arranged as to do away with the expensive bags and horse-hair mats.

The process of refining crude cotton-seed oil consists in treating it, according to its condition, with 7 to 15 per cent. of caustic soda. The lye, at a temperature of 60° F., is uniformly distributed over the surface of the oil by means of a perforated criss-cross pipe supported over the surface of the oil. During this time the oil is constantly agitated by a mechanical contrivance or by blowing in air, agitation being continued for about half an hour, and at the end of that time a sample of the oil is taken out and allowed to stand. If the black, flocculent particles of soap formed by the partial saponification of a portion of the oil by the soda lye settle, and the oil is found nearly deprived of color, the operation is then terminated. If not, the agitation is continued, more lye being added until the desired discoloration is attained. The charge of oil is then allowed to stand for 12 to 15 hours until the "mucilage," or partially saponified portion of the oil, together with the liquid excess of lye used, has settled away. The clear oil is then run off from the brown sediment, called *soap stock*, and the refining completed by washing and bleaching.

Crude cotton-seed oil is thickly fluid, twenty to thirty times less fluid than water, and has a specific gravity of 0.922 to 0.930 at 59° F. Below 50° F. it commences to separate palmitin, and it congeals between 28.5° and 26.5° F. The refined oil is of a pale straw color, and its specific gravity is from 0.923 to 0.928 at 59° F. While, as regards taste and odor, the crude oil resembles linseed oil, the refined product has a nutty flavor. Cotton-seed oil, as to its liquid portions, consists chiefly of olein and a small quantity of the glyceride of linoleic acid, and, as to its solid portions, principally of palmitin and perhaps some stearin. The non-saponifiable constituent amounts, according to Allen and Thompson, to 1.64 per cent.

The saponification number of cotton-seed oil is 191 to 196.5, and that of the fatty acids separated from it 205.9. The iodine number of the oil is 106 to 108.7, and that of the fatty acids 110.9 to 111.4. The fatty acids separated from it melt at 95.3° to 101° F. and congeal at 95° to 100.4° F.

In the United States the crude cotton-seed oil is used for lubricating purposes, as a substitute for linseed oil in the manufacture of varnish, and in the fabrication of soap; the refined oil is employed as a table oil, in the manufacture of soap, and largely for the adulteration of other oils.

The crude oil saponifies with greater ease than the refined product; this being very likely due to the former containing free fatty acids while the latter is neutral in consequence of the treatment with lye in refining. Refined oil by itself saponifies with difficulty even with strong lyes, but readily in combination with fats which saponify with ease, such as palm-kernel oil, cocoanut oil, etc. By itself it can only be gradually brought to complete saponification by weak lyes; the resulting soap has, however, the disagreeable property of being difficult to salt out, and of not completely yielding the excess of water, even with a large addition of common salt. Such soap is so soft and smeary that it can be worked with a shovel, and, on pressing in the hand, yields water. The fresh soap is white, but becomes yellow after drying, and has a peculiar, bad odor.

In Europe refined oil only is used in the manufacture of soap. It is employed for hard and soft soaps; however, never by itself, but always in combination with other fats on account of its difficult saponification and the disagreeable odor acquired by pure cotton-seed oil soaps on storing. Soaps manufactured from a large percentage of cotton-seed oil in combination with other fats also show this disagreeable odor; it can, however, be removed to some extent by boiling the oil with soda lye of 25° B. before use.

Commercial refined oil is of a sufficiently pale color for most soaps for which it is used. Where a lighter color is desired the object can be attained by treating the oil with caustic soda lye or with potassium chromate and hydrochloric acid.

For hard soaps cotton-seed oil is generally used in combination with palm-kernel oil and cocoanut oil, it possessing the property of making soaps from these oils soft and delicate. At one time cotton-seed oil was very much liked for smooth

white grained soaps, but the soap-boilers who were first in raptures over the fine results attained, had soon cause for regret when yellow stains made their appearance. These yellow stains are due to a yellow, non-saponifiable, oleaginous body in the oil, which is not destroyed by refining with lye, and not completely removed by repeated refining. In France and Italy considerable quantities of cotton-seed oil are worked in combination with peanut oil into Marseilles soap, though not to the advantage of the product.

Cotton-seed oil is also much used for soft soaps. On account of its comparatively large percentage of palmitin it cannot be used in winter for smooth, transparent soft soaps, as they readily become turbid; in summer it is, however, very suitable for such soaps, they being more solid than with the use of pure linseed oil.

For smooth yellow and smooth white soft soaps cotton-seed oil is well adapted and can be used by itself. For soap with a silver luster, so-called silver or claudin soap, it must be previously bleached.

It is also used for figged soft soaps, though great care is required in not allowing any soda to reach the soap, and using only the best high-grade potash.

The property of cotton-seed oil separating palmitin at a few degrees above 32° F. has been utilized in the United States to obtain an oil containing little palmitin, and, therefore, better adapted for the adulteration of olive oil than the natural refined oil. The separated solid fat is of a lardaceous consistency, and is brought into commerce under the name of "cotton stearin" or "vegetable stearin." According to Muter,\* it has a specific gravity of 0.912 to 0.915 at 100.5° F., is completely soluble in ether and hot absolute alcohol, and by saponification yields 95.5 per cent. of fatty acids belonging to those insoluble in water. Though the fat becomes completely liquid only at 89.5° F., the melted fat does not recongeal after cooling, but

\* Seifenfabrikant, 1882. S. 411.

forms a yellow oil, which only acquires its original consistency by long cooling at about 40° F.

Cotton stearin shows the same disagreeable properties in saponifying as cotton-seed oil, while soaps prepared from it show yellow stains and acquire a disagreeable odor on storing.

*Almond oil.*—The fat almond oil found in commerce is generally obtained from small bitter almonds, peach and apricot kernels, and fragments of sweet almonds. It is limpid, thinly fluid, slightly yellowish, nearly inodorous, of a pleasant, mild taste, and belongs to the non-drying oils. Its specific gravity is 0.915 to 0.920 at 59° F. The actual almond oil congeals at 4° F., peach-kernel oil at 0.4° F., and apricot-kernel oil at about 7° F.

Nearly all the almond oil found in commerce is cold-pressed. The almonds are crushed with edge runners, sifted and bolted, and the resulting fine meal is subjected to hydraulic pressure. The press cake is as a rule reconverted into meal and again pressed, though in doing this the mass is seldom heated. Oils of an inferior grade are prepared from defective almonds, or are adulterated. The press cake ground and finely pulverized forms what is known as *almond meal*.

The finest qualities of almond oil are used for medicinal purposes, and others, if of a good and pure taste, for mixing with table oils. Inferior qualities are in demand for technical purposes, particularly for the preparation of toilet soap. A soap from three-fourths cocoanut and one-fourth almond oil is very hard and solid and an excellent toilet soap. Almond oil can be saponified in the cold way.

*Linseed-oil* is obtained from the seeds of the flax-plant, *Linum usitatissimum* L., by pressure and the aid of heat. Ripe seed contains 30 to 35 per cent. of oil and that not thoroughly matured, less. Commercial seed yields on an average 22 per cent. of oil. Cold-drawn linseed-oil is nearly colorless; hot-pressed oil has a golden-yellow color changing to brown with age.

Linseed-oil has a peculiar odor and possesses the most dry-

ing properties of all known oils. On exposure to the air it eagerly absorbs oxygen, soon becoming rancid and thickly-fluid; in a thin layer it dries to a neutral body insoluble in ether. It has a specific gravity of 0.930 to 0.935 at 59° F., and congeals to a solid yellow mass at about—16.5° F. The fatty acids separated from linseed oil melt at from 52° to 62.5° F., and congeal at about 55.5° F. The saponification number is 189 to 195, the iodine number 132 to 180, and the iodine number of the separated fatty acids 179 to 182. The chemical composition of linseed oil has not yet been satisfactorily determined. It contains but small quantities of glycerides of solid fatty acids, palmitin and myristin, while the fluid portion which amounts to about 90 per cent. very likely consists of the glycerides of linoleic, linolenic, isolinoleic and oleic acids.

There are frequent complaints about the adulteration of linseed oil. The surest way of testing the oil as to its purity is the determination of the iodine number, that of linseed oil being the highest of all known oils. A very simple test is to expose the oil to a low temperature; if it separates solid fat at a few degrees below 32° F., or congeals, it is not pure.

Linseed oil is largely employed in Germany in the manufacture of soft soaps, and it is well adapted for both winter and summer soaps. Potash soap from pure linseed oil does not freeze even on exposure to the coldest weather occurring in this country. For summer soft soap it is recommended not to use linseed oil by itself, but to add some oleic acid or cotton-seed oil. For the transparent, pale soft soaps so much in demand at the present time the linseed oil has to be bleached. A pale oil may be used without bleaching by thoroughly boiling in a lye of 23.5° to 26.5° B. This must, however, be determined by experiment, because a pale linseed oil cannot be well defined. An oil may appear pale and clear, but when it is boiled the soap rapidly becomes darker in color, while another oil of the same appearance will give a soap that will remain pale.

A dark brownish linseed oil, which is not decolorized by lye, cannot be improved by the use of potassium dichromate and acid. Oil of a greenish tinge generally bleaches very light with dichromate and acid. Generally speaking, linseed oil saponifies with ease. Oil bleached with lye, being entirely neutral, is somewhat more difficult to saponify than crude oil or that bleached with dichromate and acid. It should further be borne in mind that a thinly-fluid oil like linseed oil requires lyes of greater causticity than an oil containing more solid constituents, as otherwise the soap turns out too soft. An advantage of linseed oil is that many distilled oleins, which by themselves do not yield a serviceable soap, can be worked in combination with it.

*Cameline oil* or *German sesame oil* is obtained by expression from the seeds of the gold-pleasure, *Camelina sativa*, Cez., or *Myagrum sativum*, L., natural order *Crucifera*. It is golden-yellow, has slight drying properties, and a peculiar taste and odor. Its specific gravity is 0.925 to 0.930 at 59° F.; it congeals at —0.4° F. Cameline oil, being produced in comparatively small quantitives, is of but little importance, though it is, well adapted for the fabrication of soft soaps which do not freeze even on exposure to great cold. Barrel soaps from cameline oil can scarcely be kept in summer, as they melt below 68° F.

*Niger oil* is obtained from the seeds of *Guizotia oleifera*, D. C., cultivated in India. Large quantities of seed are shipped from Bombay to England, where they are expressed. The oil is of a yellowish color and a mild and nutty taste and odor. It has a specific gravity of 0.924 at 59° F., thickens at 17.5° F., forms a transparent yellowish mass at 14° F., and a solid whitish mass at 5° F. In England it is much used in the manufacture of soap.

*Madi-oil* is obtained from the seeds of *Madia sativa*, Mdt., natural order *Composita*, indigenous to Chili. Experiments in cultivating the plant in France and Southern Germany proved a failure. The oil is dark yellow and has a peculiar,

though not disagreeable, odor and taste. Its specific gravity is 0.928 to 0.935; it congeals, according to Winckler, at between 14° and 12° F., and, according to Riegel, at—13° F. With soda lye it yields a firm, inodorous soap.

*Hemp-seed oil* is expressed from the seeds of *Canabis sativa*, L. Large quantities of the oil are brought into commerce from the Russian Baltic provinces. It has a strong odor and a sickly taste. When fresh it is of a greenish-yellow color, but becomes brown-yellow with time. Its specific gravity is 0.925 to 0.931 at 59° F.; it thickens at 5° F. and concretes at—16.5° F. The melting-point of the separated fatty acids is at 66° F. and their congealing-point at 59° F. The saponification number of the oil is, according to Valenta, 193.1; the iodine number, according to Hübl, 143; and that of the separated fatty acids, according to Morawski and Demski, 122.2 to 125.2. In boiling alcohol hemp-seed oil dissolves in all proportions; of cold alcohol it requires 30 parts. A solution of 12 parts in boiling alcohol, on cooling, separates stearin. Hemp-seed oil has strong drying properties. It was formerly much used in the fabrication of soft soaps which have a dark green color and can be submitted to intense cold without solidifying. The green soft soaps found at present in commerce are mostly artificially colored linseed-oil soaps.

*Sunflower-oil*.—This oil, obtained by expression from the seeds of several species of *Helianthus*, is chiefly brought into commerce from Russia. It is limpid, pale yellow, dries slowly, and, when cold-drawn, has an agreeable odor and mild taste. Its specific gravity is 0.924 to 0.926; it congeals at about 3° F. The separated fatty acids melt at 73.5° F. and congeal at 62.5° F.

At the present time the larger part of the oil is consumed in Russia itself, where the cold-drawn oil is used as table-oil, and the hot-pressed in the manufacture of varnish and soap. Considerable quantities were formerly exported and were readily taken by soap boilers and used for bar soap and soft soap.

*Corn-oil.*—Maize or Indian corn, the seed of *Zea Mais* contains 6 to 9 per cent. of oil, the latter being contained chiefly in the germ of the grain. The oil is not obtained by direct expression, but as a by-product, chiefly in the manufacture of starch and glucose. The hulls are loosened by steeping the corn for several days in water at 175° F., to which sulphuric acid has been added to prevent fermentation. The softened kernels are crushed between rollers and the broken grain is then by means of a special apparatus, called a degenerator, separated into starch, hulls and germs. The latter float upon the surface of the water and, after being removed and dried, are formed into cakes. The latter are subjected to pressure in a hydraulic press, whereby about 15 per cent. of a quite thickly-fluid oil of a pale yellow to gold-yellow color and an agreeable taste and odor is obtained. It has a specific gravity of 0.9215 at 59° F., and congeals at 14 to 15° F. to a quite solid white mass. The saponification number of the oil is 188.1 to 189.2, and that of the separated fatty acids 198.4; the iodine number of the oil is 119.4 to 119.9, and that of the fatty acids 125.0. The oil shows no absorption of oxygen and contains 1.35 to 1.55 per cent. non-saponifiable substance. It is quite soluble in acetone; only slightly so in alcohol and glacial acetic acid.

*Poppy-seed oil* is extracted by expression from the seeds of *Papaver somniferum*. It has a slight odor and mild taste; is nearly colorless or pale golden-yellow, and limpid. The oil of the second pressure is darker. Poppy-seed oil has a specific gravity of 0.924 to 0.937 at 59° F. and congeals at —0.4° F. The separated fatty acids melt at 69° F. and congeal at 62° F. The saponification number is 192.8 to 194.6; the iodine number 134 to 136.

Poppy-seed oil is chiefly used as a table oil and in oil painting. On account of its high price it is not much used in the fabrication of soap; the thick foots serve for the fabrication of soft soaps.

*Rape oil (colza oil)* is obtained from varieties of *Brassica*

*campestris*, the seeds being crushed, heated and pressed after the manner of cotton seed. The specific gravity of the oil ranges between 0.9128 and 0.9175 at 59° F., and the congealing point between 28.5° and 14° F. The saponification number is 177 to 179, the iodine number 100 to 103.6, and that of the separated fatty acids 96.3 to 99.02. Rape oil consists chiefly of the glycerides of oleic, stearic and brassic acids, and contains, according to Allen and Thompson, one per cent. of non-saponifiable substance. Its color is pale to dark brown-yellow. When fresh it is almost inodorous, but by age acquires a peculiar odor. It becomes gummy on exposure to air, but does not actually dry. It is but seldom used in the manufacture of soap, with the exception of the foots, which are frequently utilized for soft soap. They saponify with difficulty, and the soft soaps prepared from them break up at a moderately cold temperature. With soda lye, rape oil yields a crumbly soap of poor quality.

*Utilization of foots.*—Large lots of foots or sediment accumulated in oil reservoirs are frequently offered to soap-boilers. This material generally constitutes a dark and smeary, or sometimes, a lardaceous solid mass. It is suitable for the manufacture of soft soaps as well as of bar soaps, which are valuable for milling. It may also be used for rosined soaps, the strong odor which generally adheres to it being then immediately covered by that of the rosin. Foots are worked in various ways; some boil them to grain and add a small quantity of the resulting soap to each boiling of soap, while others add a small quantity of the foots directly to the soap-stock, which can be very well done, for instance, with half-boiled soap. It depends, however, more or less, on whether the foots are comparatively pure and not too old. Another method yielding an excellent product consists in boiling the foots upon strong brine until the clear oil free from scum floats on top. One thousand pounds of this oil are brought into an open kettle, together with 800 pounds of water and 60 pounds of common salt, and boiled 10 to 12 hours. The kettle is

then allowed to stand uncovered for 24 hours, when the clear oil on the top is drawn off. Beneath the clear oil is a layer of slime mixed with hulls of seed, etc. The oil retained by this is gradually separated by filling petroleum barrels half full with this slime, adding brine, and after covering the barrels exposing them to the sun. This process is still better if executed with steam.

#### FATTY ACIDS AND ROSIN.

*Fatty acids.*—The employment of fatty acids for the preparation of soap may be said to date from the manufacture of stearin. When the latter industry sprung into existence and crude oleic acid was obtained as a by-product, it was endeavored to utilize the latter primarily for the manufacture of soap. In fact, the patent granted, in 1825, to Gay-Lussac and Chevreul, for the separation of fatty acids and their application to the manufacture of candles, contains the following specification : “The liquid bodies separated are to be converted into soap.” \*

The crude oleic acid of stearin manufactories was for a long time the only fatty acid used in the preparation of soap. The fatty acids regained from the wash waters of cloth mills and other textile industries were next brought into commerce under the name of “fuller’s fat.” Later on, the high price of glycerin induced a number of stearin manufacturers to saponify neutral fats in order to obtain the glycerin, and to sell the fatty acids to the soap boiler. In this way fatty acids of palm oil, palm-kernel oil, olive oil and bone fat came into commerce. They were no doubt prepared by first saponifying the fats in autoclaves and then distilling the fatty acids with superheated steam to give them a fine white appearance. Another fatty acid, known as *rapolein* was for some time offered to soap boilers. It formed a clear, dark red, nearly inodorous oil, and was very likely prepared from residues

\* Brevets d' invention 41, p. 392.

obtained in refining oil. It was well adapted, either by itself or in combination with linseed oil, for the manufacture of summer soft soaps where no objection was made to their brown color.

The above-mentioned rise in the price of glycerin has induced many soap manufacturers to instal autoclave-saponification in order to withdraw from the fats the glycerin before working them into soap. Since, however, the installation of autoclaves is connected with considerable expense, many soap manufacturers prefer the fermentation-process of splitting the fats which can be installed at much less cost.

As regards the fatty acids obtained by the various processes of saponification, it may be said that those gained by autoclave saponification, especially those from coconut oil and palm-kernel oil, are as light in color as the original neutral fats, while other fats, such as bone fat, linseed, peanut and cotton-seed oils suffer in color so that the respective fatty acids cannot be used when a light color is of importance. Moreover all fatty acids oxidize when for some time exposed to the air, and then yield dark soaps. For this reason it is advisable to convert them as soon as possible after they have been obtained into soap.

The fermentative process of splitting of fats yields very fine fatty acids of a light color and for this reason is preferable to the older autoclave-saponification.

Twitchell's process gives very good results, fatty acids of a very high percentage and accordingly large yields of glycerin being thereby obtained. Complaints are frequently heard that soaps made from fatty acids produced by this method, become, in time, considerably darker. This is very likely due to the fact that Twitchell's instructions to work during the splitting under complete exclusion of the air are not strictly followed.

Of the above-mentioned acids it will only be necessary to discuss more fully the oleic acid, also called olein or elain, of the stearin factories, and fuller's fat.

*Olein*.—In the manufacture of stearin the fat, as previously mentioned, is saponified for the separation of the glycerin, and the resulting mixture of fatty acid is separated by pressure into a solid and a liquid portion. The cakes obtained by the first cold pressure are again subjected to a second warm pressure. The resulting solid white mass is known as "stearin," though scientifically the term is incorrect, since the product is not the glyceride of stearic acid, but essentially a mixture of stearic and palmitic acids.

The liquid portion running off by the first cold pressure is "oleic acid," which, however, contains a considerable quantity of stearic and palmitic acids, and, with previous autoclave saponification, some dissolved neutral fat. By long storing in a cool cellar, or better, by cooling with a cold-air machine, the greater portion of the solid fatty acids separates out and is extracted by a filter-press or other filtering arrangement. The finally resulting clear, oleaginous fluid forms the so-called *olein* or *elain*. In commerce a distinction is made between "saponified" and "distilled" olein, the latter being less suitable for soap-making purposes than the former. To fully understand this distinction it must be borne in mind that in the fabrication of stearin several methods are employed for the saponification of the fats, chiefly the so-called autoclave process, and acid saponification and subsequent distillation. Saponification by means of lime in an open vessel, which is the oldest method, has been entirely abandoned, chiefly on account of the cost of the large quantity of sulphuric acid required for the decomposition of the lime-soap formed. It has the further drawback of the glycerin being obtained in such dilute solution as to scarcely act upon the hydrometer. Autoclave-saponification, *i. e.*, saponification in a closed vessel under pressure with 2 to 4 per cent. of lime, or 1 to 2 per cent. magnesia is at the present time the actual source of saponified glycerin. For the separation of the lime or magnesia sulphuric acid is also used, but as a smaller quantity of these bases is employed than in ordinary lime-saponification,

less acid is required for the decomposition of the lime-soap or magnesia-soap.

In stearin factories acid-saponification is effected by bringing the heated fat, for a shorter or longer period, in contact with more or less concentrated sulphuric acid, and then boiling for some time with water and steam. After shutting off the steam the whole is allowed to rest for a while. The fatty acid mixture separates on the top; the water beneath it contains the glycerin and sulphuric acid in solution. This lower aqueous layer is removed and the fatty acid mixture again boiled with water and steam. The steam is then shut off, and the whole allowed to rest for some time. When all the fatty acids have collected on top, they are lifted off and subjected to distillation by superheated steam. By the repeated boiling with water and steam the sulphuric acid is, as a rule, completely removed from the fatty acids, and the latter are free from it when subjected to distillation. But even if they should still contain acid none of it would be found in the oleic acid as it would at all events be removed in the further process of manufacture. The opinion held by many soap boilers that the defective quality of distilled oleic acid is due to a content of sulphuric acid may therefore be regarded as erroneous. However, the question remains, What has brought distilled oleic acid into disrepute? It was formerly generally held that the defects were due to distillation, as oleic acid could not be distilled without suffering decomposition. This opinion has, however, been refuted by Bolley and Bergmann's investigations. They found that oleic acid distils over unaltered in a current of steam of 482° F., but that products of decomposition make their appearance at a higher temperature. In many stearin manufactories distillation is carried on at too high a temperature, they being forced to do this on account of the fat not being completely saponified and containing too much neutral fat. The poor quality of distilled oleic acid may, therefore, be due to products of decomposition formed under the influence of too high a temperature, and also to a

content of non-saponifiable products originating from the treatment of the fat with sulphuric acid.

Formerly, when acid-saponification was generally carried on by allowing large quantities of concentrated sulphuric acid to act a long time upon the fats, a portion of the oleic acid was always destroyed, while the remainder was so changed as to render it almost unfit for the manufacture of soap. Besides, the oleic acid always contained a considerable quantity of non-saponifiable, paraffin-like combinations (hydrocarbons). But this has been changed since experience has taught that fats can be saponified by leaving them a very short time in contact with concentrated sulphuric acid and completing saponification by boiling with water acidulated with sulphuric acid. Stearin manufacturers using this process of acid-saponification now furnish olein suitable for soap-making purposes. It may be even said that the greater part of the so-called saponified olein at present found in commerce is actually distilled, as many factories saponifying in autoclaves subsequently slightly acidulate the fatty acids obtained and submit them to distillation. With many samples of olein it is, therefore, almost impossible to decide by which process of saponification they originated, while others by their acrid odor at once indicate their production by distillation.

The best means of examining olein is to test it in regard to its saponifying ability, *i. e.*, to determine how much non-saponifiable substance it contains. This is done in exactly the same manner as examining for mineral-oils or rosin-oils (see page 69). Saponify the olein with alcoholie soda lye, mix the resulting soap with sand, evaporate the alcohol on a water-bath, wash the residue with petroleum spirit, and remove the latter from the extract by evaporating at 122° F. The residue gives the non-saponifiable, paraffin-like combinations.

A method somewhat less exact, but sufficiently accurate for technical purposes, is by directly determining by titration the quantity of saponifiable substance. Oleic acid requires for saturation 19.00 per cent. of potassium hydroxide, and as pal-

mitic acid requires 20.84 per cent. and stearic acid 18.87 per cent., we will not be far wrong in assuming 19 per cent. for olein. Mix 1 gramme of olein with 20 to 25 cubic centimeters of alcohol, color the mixture yellow with a few drops of phenolphthalein, and titrate with potash lye containing 19 grammes of potassium hydroxide to the litre, until the appearance of a red coloration. If the olein consisted of pure oleic acid 1 gramme of it would require exactly 10 grammes of this lye for saturation. Such olein is, however, not found in commerce, it containing at the utmost 97 to 98 per cent. saponifiable substance. It would, therefore, require 9.7 or 9.8 cubic centimeters of the lye so that 0.1 cubic centimeter always corresponds to 1 per cent. oleic acid. Olein containing less than 95 per cent. of saponifiable substance cannot be called good, though products with more than 20 per cent. of non-saponifiable substance are found in commerce.

Olein is used for grained soap, smooth olein soap and, in connection with linseed oil, for transparent soft soap. However, the latter having a brown color, the demand for it has much fallen off since the introduction of the light-colored soft soaps. Olein is much liked for textile soaps. Hard olein soaps are frequently used in place of olive oil soaps.

The fatty acid mixture separated from the press cakes by hot pressure in stearin factories is of a lardaceous consistency, and is generally known as *soft fat* or *margarin*. It is generally added to crude fatty acids before subjecting them to pressure, though it is sometimes worked into soap. It yields very good grained soap, but is also very suitable for smooth olein soaps.

Under the name of *white elain*, a white fatty acid of lardaceous consistency is brought into commerce from Holland. As the fat is of a pure white color it cannot have been obtained direct from the press but must have been once more subjected to distillation with super-heated steam. At one time, when the price of tallow ruled very high, it was frequently employed, in connection with cocoanut oil and palm kernel oil, in the manufacture of white grained soap. This white elain must

not be confounded with the *solid white olein* which will be referred to under fuller's fat.

*Fuller's fat*.—Under this term is understood the fatty substances recovered from the soap waters of cloth manufactories, spinning establishments, dye houses, etc. The process for the recovery of these fatty substances that is usually adopted is as follows:

The soap water is conducted into reservoirs of pine wood. Such a reservoir is about 9 feet long,  $5\frac{1}{2}$  feet wide and 5 feet deep, and holds about 1850 gallons. Sulphuric acid is added, and, in order to hasten separation, steam is introduced for from 1 to 2 hours. The quantity of sulphuric acid to be used depends chiefly on the amount of alkali in the soap water; a slight excess is, however, always allowed, since by it a quicker and more complete separation and, in consequence thereof, a more compact mass is obtained. On an average 55 pounds of sulphuric acid of 66° Baumé suffice for the complete decomposition of 1850 gallons of soap water, and will yield, according to the time allowed for draining off in the filtering basins, from 430 to 450 pounds of fatty matter. The filtering vessels consist of baskets lined with coarse hemp cloth. When the caseous, doughy mass is sufficiently drained off and has acquired the plastic consistency required for forming it into press cakes, it is wrapped up in hemp cloths and in the usual way laid between plates in a hydraulic press and pressed, at first cold, and later on with admission of steam, until the fluid contents are completely exhausted. A solid residue, about one-half of the mass, remains in the press cloths, while an equal quantity of watery fat runs into the reservoir. This is also reduced to about one-half its weight by the various operations of the refining process, so that the average yield will be about 25 per cent. of salable fuller's fat.

The crude fat in the press reservoirs has to be purified and freed from water. For the purpose of purifying, the fat is placed in copper tanks  $3\frac{1}{2}$  feet in diameter and 5 feet deep, which are fixed in iron casings. According to the greater or

lesser purity of the fat,  $\frac{1}{2}$  or  $\frac{1}{4}$  of its volume of water and 2 to 3 per cent. of its weight of sulphuric acid of  $66^{\circ}$  B. are added, and the whole is heated by direct introduction of steam to moderate boiling, which is kept up for one hour. The steam is then cut off, the mass allowed to settle for a few hours, and the lower turbid and slimy stratum drawn off. The liquid running off is replaced by an equal quantity of pure water, and the whole heated to moderate boiling in order to remove adhering sulphuric acid. The whole is next allowed to settle for 12 hours. The watery layer is then removed and the clear mass of fat drawn off. The fat thus obtained still contains a considerable quantity of water, and is freed from it by bringing it into a kettle provided with a copper or iron coil through which steam circulates.

The fat thus obtained forms a thickly fluid, oily mass of a brown to black color and disagreeable odor. Abroad it is to a considerable extent used in the manufacture of soap, though never by itself, but always in combination with other fats, especially with palm-kernel oil and rosin. It is much liked for textile soaps. It readily saponifies with lye of  $18$  to  $24^{\circ}$  B. It must, however, be strongly salted out, and it is best to boil it upon a second water, whereby it yields a good grained soap with fair washing power.

Fuller's fat consists chiefly of fatty acids, and can readily be distilled with superheated steam. The fat thus refined is commercially known as *solid white olein*. It readily saponifies with lye of  $20$  to  $25^{\circ}$  B., and yields a nice, firm grained soap of a silvery luster and good odor.

*Working fatty acids.*—The use of fatty acids for soap is based upon a much more simple chemical process than that which takes place in the saponification of neutral fats, since no previous splitting into glycerin and fatty acids is required, and the fatty acids possess the property of expelling the carbonic acid from alkaline carbonates and combining with the latter to soap. Hence with the use of fatty acids for the manufacture of soap, the preparation of lye can be saved and saponification effected with solutions of alkaline carbonates.

The prejudice which formerly existed against saponification of fatty acids with alkaline carbonates has to a great extent disappeared, and this method—the so-called *carbonate saponification*—is coming more and more into use, since by it can be prepared soaps just as good in appearance and quality as those made with the assistance of caustic lyes. The process presents no difficulties whatever, especially where steam is available, although, with some experience, it can also be carried on with a direct fire. An inconvenience in the latter case is that the quantity of stock in the kettle must at least be one-third less than when boiling with caustic lye, since, on account of the escape of carbonic acid, a considerable allowance must be made for the rising of the stock in the kettle. However, the principal drawback is that the fatty acids always contain more or less neutral fat, a chemical examination being therefore required, which few soap boilers can or will undertake, and working without knowing the content of fatty acid and neutral fat may later on lead to disagreeable results.

*Carbonate saponification* is executed as follows: The quantity of alkaline carbonate, no matter whether soda ash or potash, previously accurately calculated, is dissolved in water and heated in the kettle to boiling. The fatty acid, previously liquefied, is then allowed to run in, the carbonate solution being kept constantly boiling. The fatty acid immediately combines with the alkali, whereby carbonic acid is liberated and escapes. This liberated carbonic acid causes vigorous foaming and rising of the soap mass. Hence the admission of fatty acid has to be so regulated that the soap cannot run over. Such overrunning will not occur if only so much fatty acid as will immediately be converted into soap is introduced. In especially critical moments the admission of fatty acid is interrupted till no more unsaponified particles of it are noticed upon the surface of the soap. Overrunning takes place most readily when the last remnants of fatty acid come to be saponified. In carbonate saponification it is advisable to employ a checking or curbing apparatus, it affording better protection

against the soap running over than the use of a hand crutch. In the construction of such an apparatus a piece of gas-pipe about 20 inches longer than the upper diameter of the kettle serves as a shaft. To the center of the shaft are secured, opposite to each other, two wings of stout sheet-iron, each about 6 to 8 inches wide. Two bearings screwed one opposite to the other to the kettle serve for the reception of the shaft. The apparatus is revolved by means of a crank secured to the end of the shaft projecting over the edge of the kettle. Where power is available the crank is replaced by a loose and a fast pulley. By the use of such an apparatus the soap when rising in the kettle can readily be checked. However, this rising decreases considerably when the last portion of fatty acid has been saponified. Nevertheless boiling has to be continued for some time longer to give the carbonic acid which is liberated a chance to escape, it being absolutely necessary that it should be completely expelled, as otherwise soft soaps are formed. During this time the soap becomes constantly heavier and thicker, and gradually commences to fall. The carbonic acid in escaping forces its way with violence through the soap, whereby particles of the latter continually spurt around and partly fly from the kettle. When the soap has entered this stage and lies thick and heavy in the kettle without being able to rise again, it may be supposed that all the carbonic acid has been expelled and that further boiling may be continued with the addition of the quantity of caustic lye required for the neutral fat which may have been present in the fatty acid. After the addition of this lye normal boiling again commences at once, and the previously heavy and turbid soap becomes darker and pasty, as well as more fluid. It again commences to rise, can now be fitted and regularly finished like soap boiled from neutral fats.

The calculation of carbonates for fatty acids is made according to the following rules:

Not taking into account the fatty acids of palm-kernel and cocoanut oils, which have a very high neutralization number,

100 lbs. of 100 per cent. fatty acid require for their saturation about 20 lbs. of 100 per cent. ammonia-soda ash. However, as commercial ammonia-soda has only about 98 per cent., there would be still wanting for 100 lbs. of pure fatty acid  $20 \times 0.2 = 4$  lbs. for complete saponification. But in order to be more successful with the soap it is advisable to leave this small difference out of consideration and to make up this small missing quantity of soda ash with caustic lye. It is therefore necessary to know the content of neutral fats in the fatty acids to be worked so that, based upon the results obtained, the quantities of soda ash and caustic lye required for saponification can be calculated.

This may be explained by a few examples. Since with all the methods of splitting fat known at present, up to 95 per cent. of free fatty acid is at the utmost obtained, it will always be necessary to ascertain the results of each splitting previous to boiling in order to make correct calculations. By taking first the above-mentioned result of 95 per cent. of free fatty acid and 5 per cent. of neutral fat, the following calculation results: 95 per cent.  $\times 20 : 10 = 19$ . Hence 100 lbs. of 95 per cent. fatty acid required 19 lbs. of soda ash for saponification. The 5 per cent. of neutral fat contained in the fatty acid requires, on the other hand,  $2\frac{1}{2}$  lbs. of caustic soda lye of 40° B. Suppose 5000 lbs. of such fatty acid are to be saponified, the calculation is as follows:  $50 \times 19 = 950$  and  $50 \times 2.5 = 125$ . Hence the 5000 lbs. of 95 per cent. free fatty acid require for saponification 950 lbs. of 98 per cent. soda ash and 125 lbs. of caustic soda lye of 40° B.

For a 90-percent. fatty acid to be worked the calculation is as follows:  $90 \times 20 : 100 = 18$ . Hence 100 lbs. of 90 per cent. fatty acid require for saponification 18 lbs. of soda ash and  $10 \times 0.5 = 5$  lbs. of caustic soda lye of 40° B. Hence the above-mentioned 5000 lbs. require  $15 \times 18 = 900$  lbs. soda ash and  $50 \times 5 = 250$  lbs. of caustic soda lye of 40° B.

For 85 per cent. fatty acid the calculation is as follows:  $85 \times 20 : 100 = 17$ . Fifteen per cent. fatty acid equal  $15 \times$

$0.5 = 7.5$ . Hence 100 lbs. of 85 per cent. fatty acid require for saponification 17 lbs. soda ash and 7.5 lbs. caustic soda lye of  $40^\circ$  B. Referred to the above-mentioned 5000 lbs. the result is  $50 \times 17 = 850$  lbs. and  $50 \times 7.5 = 375$  lbs. Hence 5000 lbs. of 85 per cent. fatty acid require for saponification 850 lbs. soda ash and 375 lbs. caustic soda lye of  $40^\circ$  B. These examples are sufficient to show how the alkalies have to be calculated for the fatty acid in accordance with its content of actual fatty acid and neutral fat.

For fatty acids from palm-kernel oil, which have a neutralization number of 250 to 265, 20 lbs. of soda ash for 100 lbs. of fatty acid are not sufficient. Since 56 calcium hydrate, upon which the saponification number, relatively the neutralization number, is based, corresponds to 53 sodium carbonate, we arrive at the proportion,  $56 : 25.0 = 53 : x$ , whence follows that for 100 lbs. of palm-kernel oil fatty acid at least 24 lbs. of soda ash are required.

Special attention must be called to the fact that the calculations given above are purely theoretical and show only how much caustic soda lye in addition to soda ash is absolutely required. For practical work a larger consumption of caustic soda lye will, as a rule, be necessary in order to attain a vigorous fitting of the soap. To the novice in carbonate saponification it may also happen that saponification with alkaline carbonates is not accomplished in an entirely regular manner, either because lumps are formed in running in the fatty acid, or the carbonic acid had not sufficient time or chance to escape. In both cases the calculated quantity of caustic soda lye will not be sufficient, as the soap must by all means be fitted to a perceptible touch. Hence the novice has to be doubly cautious but, on the other hand, he should not be guided by false principles in calculating the caustic soda lye if, by neglecting some detail or by faulty boiling, the quantity of soda lye does not suffice. At all events care has to be taken that the finished soap receives an ample and sufficiently strong fit, even if more caustic soda lye is used than required accord-

ing to calculation. The process of carbonate-saponification is about the same as that described for boiling all kinds of fatty acids whether from tallow, or palm kernel, cocoanut, cotton seed, peanut oils, etc., and the work can therefore be always carried on according to the same general plan. It may be mentioned that fatty acids of any kind should not be stored in iron holders and not be handled in iron ladles or pots, as they possess to a high degree the power of attacking and dissolving iron and in consequence acquire very rapidly a red, and eventually a brown, color. Barrels in which fatty acids are kept become leaky in a shorter or longer time, and for this reason, the fatty acids should be used as soon as possible after splitting. On account of the difference in price between soda ash and caustic soda, carbonate-saponification is of advantage only for the production of hard bar-soaps. However, its application to the manufacture of soft soaps from fatty acids would render the product more expensive since electrolytic potash lye is cheaper than pearl ash. Hence in boiling soft soaps from fatty acids the procedure is as follows:

For every 100 lbs. of fatty acid 40 lbs. of electrolytic potash lye of 50° Bé. are allowed and, according to the season of the year, 5 to 6 lbs. of pearl ash for smooth soft soap, and 10 to 12 lbs. for natural fig soap. The caustic potash lye together with the pearl ash previously dissolved in water is made up to 28 to 30° Bé., when working with direct steam, and to 26 to 27° Bé. when only an open fire is available. When the lye is heated to boiling, the fatty acid is allowed to run in; saponification takes place immediately without violent rising in the kettle. The soap gradually enters into combination and, if there has been the correct proportion between lye and water, finally lies, fitted and evaporated, in the kettle. It may eventually happen that it has to be somewhat more evaporated or the fit corrected. It may besides be subsequently ground and filled like soap from neutral fats.

It remains to add a few words regarding the boiling of hard soaps. For white-grained soaps the same kind of stock is used

as for neutral fat soaps ; care has, however, to be exercised with the supplementary fats, as after splitting they always turn out somewhat darker, while, as regards color, palm-kernel oil splits best. This also applies to Oranienburg soap, but for the rosin used in it, 12 lbs. of soda ash for every 100 lbs. have to be calculated. The boiling of Eschweg soap by means of carbonate-saponification presents at first some difficulties. They can be best overcome by first boiling from the entire stock a salted-out grain. The grain free from lye is then again contracted by means of water and about 4 lbs. of soda ash for every 100 lbs. of stock, till a pasty soap is in the kettle, which can be boiled and filled exactly like one from neutral fat. Generally speaking, all kinds of paste-soaps can, as regards carbonate-saponification, be most easily prepared according to this process.

*Rosin* is the residue from the distillation of oil of turpentine from crude turpentine. The crude oleo-resin or true turpentine is an exudation from the barks of various conifers. In regions rich in pine forests the trees are "boxed," *i. e.*, excavations are made into the trunks of the trees about six inches or more above the roots, and the crude turpentine exuding is collected in barrels and transferred to stills. The grade of rosin depends first, upon the quality of the turpentine, and, second, upon the skill in distilling. "Virgin turpentine," if skillfully distilled, will yield what is known as "window-glass rosin," of which there are two or three grades. If by any means water gets into prime rosin, it becomes opaque. This accidental addition of water must take place after the rosin has been drawn off from the still.

"Yellow-dip turpentine," which is the running of the second and subsequent years from the trees, yields the medium grades of rosin, while the "scrapings," *i. e.*, the inspissated gum from the tree facings, yield an inferior rosin, from very dark to almost black.

Common rosin is very brittle, has a glassy luster and conchoidal fracture ; its specific gravity ranges between 1.045 and

1.085 at 59° F. The melting point varies very much and depends on the content of rosin oil. Some varieties become soft at 158° F. and become semi-liquid in boiling water; others melt at 211° to 212° F., and some only at 248° to 284° F. Rosin, however, does not melt to a clear fluid. It is insoluble in water, but readily soluble in alcohol. The alcoholic solution shows an acid reaction; the acid can be accurately neutralized by caustic alkalies with the use of phenolphthalein as indicator.

The principal constituent of rosin is abietic acid ( $C_{10}H_{16}O_5$ ). It contains in addition the anhydride of this acid, and perhaps other acids in smaller quantities, and always varying quantities of non-saponifiable substance consisting of hydrocarbons, which are formed by partial decomposition of the rosin in distilling.

The most important applications of rosin are in the manufacture of varnishes, lacquers, cements, brewers' and bottlers' pitch, grease for wagons and machinery, and in the preparation of soap. In the latter industry it is employed in the manufacture of hard and soft soaps, though never by itself, but always in combination with fats. Soda soaps of pure rosin never become hard, and potash soaps from pure rosin do not acquire the characteristic consistency which is demanded from a good quality of soft soap.

Rosin being an acid it saponifies readily and rapidly, and the soaps prepared with it are distinguished by ready solubility and a good lather. Formerly it was much used in combination with tallow and palm oil for grained soaps; at the present time it is however chiefly employed in combination with palm-kernel oil, cocoanut oil, bone-fat, olein, and cotton-seed oil.

Rosin finds a further and important application in the manufacture of the various smooth and artificial fig soft soaps. The rosin—about 5 to 15 pounds to 100 pounds of oil—is either at once brought into the kettle together with the oil and saponified with it, or it is added together with the required lye of 30° Bé. to the finished soap when boiling up, and

combined with it by crutching. The latter method, which is chiefly used and can be particularly recommended for glycerin soft soap and similar varieties, gives paler soaps and a somewhat larger yield, though refitting is necessary after the addition of the rosin. On the other hand, by boiling the rosin with the fat, the soap is somewhat darker and the yield somewhat less, but refitting being not required, the work is accomplished more quickly and with greater surety. By an addition of rosin, pure soft soaps are rendered cheaper, give a better lather, and become more lustrous; they are, however, considerably softer, and it is, therefore, necessary to use about 20 to 30 per cent. of soda lye of about 24° Bé., the quantity depending on the season of the year; more being used in summer and less in winter.

It is sometimes desirable to have a very pale rosin. This can be obtained by bleaching. Melt the rosin in a kettle and allow to stand till all the dirt has settled on the bottom which will require about half an hour. The clear rosin is then brought into another kettle and to each 100 lbs. of it are added 20 lbs. of common salt solution of 9° Bé. The whole is then boiled for one hour when the fire is reduced. As soon as ebullition ceases the rosin settles on the bottom, while the salt liquor separates as a brownish fluid on the top. This salt liquor is drawn off, fresh salt water added and the whole again boiled. If the rosin is not sufficiently decolorized the operation is repeated for the third time.

The principal supplies of rosin come from the United States and France. The American grades of rosin are indicated by letters and the quality is the finer the further its letter is removed from A. The grades are as follows:

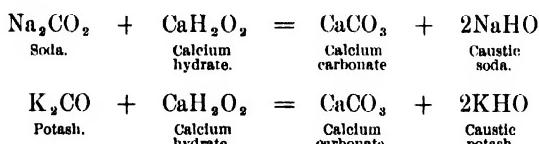
- |                   |                     |
|-------------------|---------------------|
| A. Black.         | I. Good No. 1.      |
| B. Common strain. | K. Low Pale.        |
| C. Strain.        | M. Pale.            |
| D. Good Strain.   | N. Extra Pale.      |
| E. No. 2.         | W. G. Window glass. |
| F. Good No. 2.    | W. W. Water White.  |
| H. No. 1.         |                     |

## CHAPTER VI.

### ALKALIES.

By the term "alkalies" are designated the oxides of a small group of metals which are distinguished by being lighter than water, oxidizing readily in the air, and at an ordinary temperature, decomposing water, with the development of hydrogen gas. The oxides of the metals—the alkalies—are the strongest bases known; they combine with water to hydrates, forming the so-called caustic alkalies. These have a caustic, lye-like taste, destroy the skin and all organic tissues, and are readily soluble in water. Their solutions color reddened litmus tincture blue, the coloring matter of violets and roses, green, and that of turmeric, brown; they show, as it is termed, an alkaline reaction. From the air these hydrates absorb water and carbonic acid.

Two of these alkaline hydrates are extensively used for technical purposes, potassium hydrate ( $\text{KHO}$ ) or caustic potash, and sodium hydrate ( $\text{NaHO}$ ) or caustic soda. The usual method of converting the alkaline carbonates into alkaline hydrates is by means of slaked lime (calcium hydrate,  $\text{CaH}_2\text{O}_2$ ). By bringing together solutions of alkaline carbonates with slaked lime a conversion takes place by the carbonic acid of the alkali combining with the calcium oxide to calcium carbonate, which, being insoluble in water, falls to the bottom, while the alkali combines with the hydrate of the lime and remains in solution:—



Below the manufacture and properties of the various kinds of alkaline carbonates and caustic alkalies will be briefly described, commencing with soda, which, next to common salt, is the most important of all mineral salts.

#### SODA.

The soda brought into commerce is known, according to its derivation, as (1) natural soda, and (2) artificial soda.

1. *Natural Soda*.—Sodium carbonate is widely distributed in nature as a constituent of many minerals and dissolved in many waters, thus in the soda lakes of Hungary, Egypt, Central Africa, and in the waters of the geysers of Iceland. It is occasionally found native as an efflorescence on the soil, for instance, on the steppes between the Black and Caspian Seas, on the alkali plains of North America, in Mexico, South America, etc. Extensive deposits of the chloride, sulphate and carbonate of soda are found at many points in the arid regions of the United States. These deposits occur in the desiccated beds of many ancient lakes in Nevada, Arizona, Western Utah, and portions of California and New Mexico. There are certain lakes, also, which are valuable brines.

Formerly the principal source of commercial soda was the ashes of plants growing on salt plains, near salt springs, and on the border of the sea. Plants growing in the sea itself are not suitable for this purpose, as they contain but a small quantity of alkaline carbonates, and they are principally worked on account of their content of potassium sulphate and potassium chloride, and especially of iodine. The preparation of soda from the actual soda plants, which are found along the borders of the sea to within a certain stretch into the interior of the country, is very simple, consisting only in the incineration of the plants. The industry was, and is partially at the present time, carried on on the coasts of Scotland and Ireland, but chiefly on the Mediterranean, in Sicily and Sardinia, and on the Spanish coast in the province of Valencia, in Morocco and in Armenia, and the South Russian steppes.

The best soda of this kind was the Spanish, and "barilla," the name under which it is known, was given to all vegetable sodas; even the artificial soda was first known in England as "British barilla." Barilla is also brought into commerce as Alicante, Carthagena, and Malaga soda. It forms a hard and compact mass of a dark ash or gray-blue color, and contains from 25 to 30 per cent. of sodium carbonate. It is hard and difficult to pulverize and has a sharp alkaline taste. It is obtained from plants specially cultivated for the purpose.

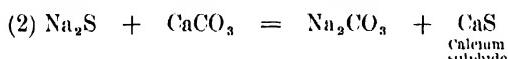
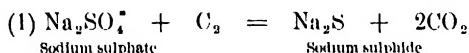
Vegetable soda, being only caked vegetable ash not purified by lixiviation, contains all the inorganic constituents of the plants. Hence, when treated with water, there always remains a considerable residue of combinations of lime, iron, etc. The portion soluble in water contains, besides sodium carbonate (and potassium carbonate), alkaline sulphates and chlorides.

At the present time the natural soda has been almost everywhere replaced by the artificial product, it being only used in the countries of its production.

2. *Artificial soda*.—During the last century the soap industry of France had become so extensive that twenty to thirty millions of francs were annually sent to Spain and other countries for vegetable soda. In 1793, when France, by its war with England, was excluded from intercourse with other nations, and, therefore, dependent on its own resources, the want of potash and soda was much felt in the soap industry. A commission was appointed to examine methods for the fabrication of soda. Of the various processes communicated, the one proposed by Nicolas Leblanc, to manufacture soda from common salt, was considered the only practical one. The industrial process was exposed with such precision that since that time—more than a hundred years—very few changes have been made.

*Leblanc's soda process* consists in heating common salt with sulphuric acid, whereby sodium sulphate and hydrogen chloride are produced. The sodium sulphate, technically

called *salt cake*, is then mixed with small coal and limestone, and again heated in order to convert it into sodium carbonate, a change which may be represented by the two equations:



The resulting mixture of sodium carbonate and calcium sulphide, technically called *black ash*—being black from the presence of coal—is leached with water to dissolve the sodium carbonate and leave the calcium sulphide (tank waste). The liquor is evaporated to crystallize the sodium carbonate (soda crystals).

Leblanc's process has been almost completely superseded by the

*Ammonia-soda process or Solvay's process.*—This process which was first brought into practical use by Solvay (1863) depends on the fact that when solutions of sodium chloride and ammonium bicarbonate are mixed, double decomposition occurs, resulting in the formation of ammonium chloride and sodium bicarbonate, the latter being precipitated on account of its sparing solubility in solutions of the former. Common salt solution is first saturated with ammonia and then with carbonic acid. Ammonium bicarbonate is formed which with the common salt is converted into sodium bicarbonate and ammonium chloride. By heating, the sodium bicarbonate is decomposed to sodium carbonate and carbonic acid. The carbonic acid thereby generated is again used for the formation of ammonium bicarbonate. From the ammonium chloride solution obtained in the beginning of the process the ammonia is recovered by heating with lime. The process is thus a continuous one in which, independent of the losses of ammonia, only common salt and a portion of the carbonic acid have to be introduced, soda alone being taken out. Only the lime required for the regeneration of the ammonia as well as

the chloride from the common salt are lost. The entire process appears very simple, but very complicated apparatus is required and it takes considerable capital to start an ammonia-soda plant.

Soda prepared by the ammonia process is very pure, it being absolutely free from caustic soda, sulphur combinations, and iron. It can be obtained without difficulty, so as to show 98 to 99 per cent.

*Production of soda by electrolysis.*—The electrolytic process of obtaining caustic soda and carbonate of soda from common salt solutions presented at first many difficulties. However, they have to a great extent been overcome and the process is making rapid headway in Europe and in this country. The Castner-Kellner process especially has proved successful. In the electrolytic process sodium separates on the cathode and then forms with the water, sodium oxide (and hydrogen). For the purpose of obtaining soda the oxide is precipitated as sodium bicarbonate by the introduction of carbonic acid. The sodium bicarbonate is then decomposed by heating into soda and free carbonic acid.

*Cryolite soda* may here be mentioned, though it is now of little importance. It is obtained from cryolite, a mineral occurring in Greenland, by heating the pulverized mineral with  $1\frac{1}{2}$  times its quantity of lime to redness, whereby, under the development of carbonic acid, calcium fluoride and sodium aluminate are formed. The calcined mass is lixiviated ; the calcium fluoride remains behind, while the sodium aluminate passes into solution. By the introduction of carbonic acid, generated by the combustion of coal, into the solution, the sodium aluminate is converted into alumina, which separates, and into sodium carbonate, which remains in solution. The soda solution is concentrated by evaporation, the larger portion of the soda crystallizing out in from eight to ten days. The alumina separated is not pure, it constituting a mixture of 45 per cent. alumina, 20 per cent. sodium bicarbonate, and 35 per cent. water ; it is, as a rule, further worked for alum.

*Sal soda, washing soda, crystallized soda*, though containing 37 per cent. of sodium carbonate, is preferred for scouring wool, for many other industrial purposes and for domestic use, largely because of its freedom from caustic soda. It is usually prepared by recrystallizing the solution of soda ash, which should be freed from sulphide and should contain not more than 2 per cent. of caustic soda.

The manufacture of sal soda may be suitably combined with the manufacture of soap. In smaller establishments any kettles temporarily not in use may be employed. For the manufacture on a large scale space is the chief requisite, and it is desirable that the room for the installation of the crystallizing tanks should be as airy as possible so that, according to requirement, a current of air can constantly pass through it from two opposite directions, more rapid crystallization of the soda being thereby effected. For this reason the room should, if possible, have a northern exposure.

The solution of the soda-ash may be successfully effected either upon a direct fire or by steam in an ordinary soap kettle. Soda-ash dissolves best at a temperature of from 120° to 122° F. When the water has been heated to that point, the soda-ash is gradually added. On account of the caking properties of the soda-ash, the fluid has to be constantly agitated during this operation by means of a crutch or a mechanical stirrer till the required degree of concentration has been reached. This point is recognized by further additions of soda falling without being dissolved to the bottom of the kettle, and also by the solution showing even in a hot state a density of 36° B. The soda solution is now allowed to rest till the next morning for the coloring matter and impurities to settle. The solution, which is now no longer turbid, but clear as water, is then transferred from the kettle to the crystallizing tanks, in which, according to the size of these vessels, the formation of crystals takes place in a shorter or longer time. Various ways may be adopted for transferring the soda solution from the kettle to the crystallizing tanks, ladling

being the most primitive and time-consuming. Pumping takes less time, but has the drawback of the sediment in the kettle being also brought into the crystallizing tanks, and dirty crystals will be the result. In installing a new plant it is advisable to locate the dissolving tank at such a height that its bottom is at a level with the upper edge of the crystallizing tank. The bottom of the dissolving tank is fitted with a discharge pipe for drawing off the sediment, and about four inches above the bottom is another discharge pipe for the clear solution. By a pipe emptying into an iron conduit the clear solution can then be distributed into any tank desired.

The crystallizing tanks are of various sizes, with a capacity of from 100 to 10,000 lbs. The smaller tanks are, as a rule, tinned, and can be readily emptied by inverting them and pouring hot water over them, which causes the block of soda to drop out. The larger tanks, on the other hand, have to be emptied by means of a hammer and chisel, but yield a product of greater purity and whiteness, as well as in larger pieces. They are generally four-cornered, of greater width than depth, and provided with a bottom running obliquely from both sides towards the center. On the deepest point is a hole suitable for the reception of a piece of gas pipe terminating in a cone. This cone serves for closing the hole, while the pipe itself projects about  $1\frac{1}{2}$  feet above the edge of the tank. This pipe serves for the purpose of running off the mother-liquor remaining after crystallization is complete. For this purpose the tanks are set upon brick pillars. When the mother-lye is to be discharged, hot water is poured from above into the gas pipe, whereby the soda on the outside of the pipe is melted so that the latter can be drawn up, thus allowing the mother-liquor to run off through the hole in the bottom into reservoirs, or it may be conducted by means of pipes to be utilized in the next operation. The tanks are allowed to stand for one or two days for all the mother-liquor to drain off. The superficially dry soda is then, as previously mentioned, broken out with a hammer and chisel and, before packing, has to be centrifuged to get it sufficiently dry.

The time required for crystallization varies, as above mentioned, according to the size of the tanks, and besides, the temperature of the air exerts considerable influence. In the hot summer months crystallization is incomplete, and even no crystals at all may be formed, so that it is actually advisable to stop operations entirely during this time. On the other hand, in winter, solutions in tanks up to 10,000 lbs. capacity are, according to the prevailing temperature, converted into crystals in ten to fourteen days.

Ammonia-soda ash of 98 to 100 per cent. is now largely used for the manufacture of sal soda, but it does unfortunately not possess the property of yielding crystals of sufficient solidity, they being of a loose structure and readily crumble and fall to pieces. In order to obtain crystals of the desired hardness and solidity it is necessary in dissolving the soda to add small quantities of Glauber's salt. As the latter is cheaper than soda ash, an addition of it would therefore tend to cheapening the finished product. This, however, is only apparently the case, as the quantity required is very small and has to be limited to what is absolutely necessary, because Glauber's salt possesses the property of dissolving particles of iron from the dissolving and crystallizing tanks, which causes a red-brown coloration of the soda crystals, rendering them unsalable. Furthermore, only small portions of the Glauber's salt pass into crystallization, the greater portion of it remaining in the mother-liquor. By repeatedly reusing the latter and the continual addition of Glauber's salt, the liquors become then so rich in the latter that the red coloration of the soda finally makes its appearance, the yield decreasing at the same time, while the residue of mother-liquor constantly becomes larger. Hence the addition of Glauber's salt can only be considered as a necessary evil, and it should be stopped so soon as the mother-liquor is noticed to become rich in it. For the first solutions 2 to 3 per cent. of Glauber's salt at the utmost suffices, and if the first mother-liquors are again utilized, the addition of it should be reduced from 2 to 1 per cent. and be

entirely stopped when the quantity of mother-liquor becomes larger; only when the latter is again normal, additions of 1 per cent. Glauber's salt may be proceeded with. When the red coloration of the soda crystals is first noticed it is too late to stop the addition of Glauber's salt, because the coloration will naturally be more pronounced in the crystallizing tanks coming into operation later on, since the action of the salt upon the iron becomes constantly stronger. The first traces of coloration usually appear in the form of reddish-brown drops adhering to the uppermost crystals. The latter are frequently covered with such a coat while the interior is still colorless. The addition of 1 $\frac{1}{2}$  to 2 ozs. of chloride of lime to every 220 lbs. of soda to be dissolved has to a certain extent proved to be a precautionary measure, but if continually used the soda readily acquires a chlorine odor which is objectionable.

The crystallizing tanks are filled nearly up to the edge, and strips of hoop-iron are laid in all directions across the tank so as to touch the surface of the liquor. On this frame of iron crystallization commences, and assisted by it, a complete crust of crystals is soon formed, the latter growing downwards in the liquor and frequently attaining a length of more than 11 inches; these yield the best product. The crystals forming on the sides of the tank, which have to be detached with chisels, are not so fine, and the poorest are those formed on the bottom. To prevent contamination with iron the tanks must be kept bright and free from rust; sometimes they are provided with a coat of paint.

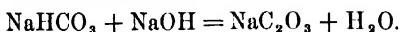
In manufacturing on a large scale the soda crystals are dried by means of a hydro-extractor, while in smaller establishments spreading the soda upon hurdles or a clean floor will in most cases suffice.

The mother-liquors from the crystals always contain sodium carbonate and the more of it the higher the temperature during crystallization has been. In addition they contain all the caustic soda and the greater part of the sodium chloride and sodium sulphate which, as a rule, only to a slight extent

crystallizes together with the soda. The mother-liquors are evaporated in pans to the consistency of paste and then calcined in reverberatory furnaces; they yield a very white, but low-grade, soda salt.

Sal soda is frequently adulterated with the much cheaper Glauber's salt. Such adulteration can be readily detected as follows: Dissolve a small quantity of the soda, acidulate with hydrochloric acid, and mix with barium chloride solution; a thick white precipitate indicates the presence of Glauber's salt.

*Fine soda.* Under this name a variety of soda consisting of very small crystals has lately come into favor. It has the advantage over ordinary sal soda in pieces of dissolving with much greater ease and allowing of better packing. According to B. Cordes it is prepared as follows: Dissolve in a kettle furnished with a stirrer high-grade ammonia-soda to a liquor of 36° B.; two parts water and one part soda being required for this purpose. The water is heated by steam to 77° F. In dissolving the soda is spontaneously heated to from 104° to 113° F., except when it has absorbed water by having been stored in a damp place and has become hard; in this case heating has to be somewhat assisted with steam. The solubility of soda is greatest at the above mentioned temperature, it dissolving to a density of about 38° B. It is however of greater advantage to dissolve only to a density of only 36° B. as the dirt then settles better. The solubility is also frequently lessened by the presence of sodium bicarbonate ( $\text{NaHCO}_3$ ). In the presence of larger quantities of this salt, the accumulation of which is due to the repeated use of the precipitates and mother-liquors, a very whitish, pasty liquor is formed which does not settle. It then becomes necessary to add caustic soda lye whereby the sodium bicarbonate is converted into ordinary soda according to the equation:



It would, however, be of great advantage to from time to time remove the sediment and mother-liquor and use them

for the saponification of fatty acid. In case the solution does no longer yield a white soda after the sediment has been several times used, a small quantity of calcium chloride solution has to be employed.

The solution must thoroughly settle by standing over night. The dirty top cover must then be carefully removed or the layer between scum and dirt is run off into the crystallizing tank. This has, however, to be done in small portions, as the result desired is only attained by rapid cooling. For crystallization wide, shallow tanks provided with stirrers and openings which can be closed are used. Rapid cooling is effected by an exhauster, and the formation of large crystals is prevented by the stirrer. When crystallization is complete the openings in the bottom of the tank are opened and the crystals are conveyed by the stirrer into a reservoir beneath the tank. The openings are then closed and a fresh charge of liquor run in, the operation being repeated so long as liquor is on hand. The crystals in the reservoir are freed from moisture in a centrifugal for five to ten minutes, when the finished product may be packed in bags and at once sent to market. The mother-liquor collects in a reservoir, and by means of a steam-jet pump is conveyed to the dissolving tank to be again utilized for the next solution.

*Caustic soda.*—Under this name is understood a product consisting chiefly or entirely of sodium hydrate. There is also found in commerce a soda lye which is shipped in carboys. For many evident reasons the trade in this article is not very extensive, consumers generally preferring to prepare it themselves from soda ash and lime. The manufacture of *solid caustic soda* was originally introduced into England in 1844 by a German named Weissenfeld. The actual commencement of its manufacture on a large scale dates, however, from about 1853, when William Gossage took out a patent, which, besides other improvements in the manufacture of soda, included the gaining of caustic soda from soda lyes by concentration and without the use of lime. The caustic soda first brought into

commerce was colored blue, green, yellow, red, etc., it being only in 1860 that Ralston succeeded in producing the white product by heating the caustic soda to a higher temperature than was formerly customary *i. e.*, to a temperature at which the iron separates as oxide and the clear caustic soda is left standing over it.

Thompson, in 1857 introduced the mode of packing caustic soda in sheet-iron drums, which is still in general use. Previous to that time the caustic soda was poured out upon iron plates, when cold broken up into pieces, and packed in barrels. This was very unpleasant work in the execution of which the soda could reabsorb much water and carbonic acid. For the manufacture of caustic soda, soda ash was formerly in many localities dissolved and made caustic by lime. This method being now too expensive has been almost entirely abandoned, and the crude lye obtained in the lixiviation of crude soda is now treated with lime. The lye, before being made caustic, must be thoroughly clarified and a good quality of lime has to be used. The crude lye is diluted with water to a density from  $11^{\circ}$  to  $13^{\circ}$  B., as more concentrated solutions cannot be made completely caustic. It is safe to go as high as  $13^{\circ}$  B., and in doing so less water has to be evaporated. Many factories even go as high as  $15^{\circ}$  B., whereby only about 92 per cent. of the soda is made caustic, but a saving in coal is effected. The lye is brought to boiling and the lime added with constant stirring. It is best to use quick lime, as it is immediately slaked on being added, and the heat developed thereby saves steam. A skilled workman can judge from the manner of boiling, the color of the liquid, and other indications when a sufficient quantity of lime has been added; this can, however, also be tested by filtering a sample and adding some sulphuric or hydrochloric acid, whereby no effervescence must take place. The entire charge can then be either drawn off into a settling vessel, or it is allowed to settle in the pan itself, which requires about half an hour. The clear lye is then gradually drawn off and pumped into vessels for complete

clarification. Generally a second operation is carried on in the pan without removing the lime-slime. The pan is again charged with crude lye and water, the contents brought to boiling and made caustic, somewhat less lime being required than for the first charge, since some caustic lime remains in the slime. After the clear lye is drawn off, the lime-slime is stirred with pure water to a thin paste and brought upon especially constructed filters. The filtrate serves for the dilution of the crude lye. The remaining lime-slime is generally used as a substitute for a portion of the quick lime for a fresh soda mixture, it being well adapted for this purpose, as it is in a finely divided state and, besides the soda contained in it, is thus utilized.

The caustic soda obtained in this manner is still in a state of great dilution. Its concentration is an important task, as it is necessary to save as much fuel as possible, and is effected in cast-iron or wrought-iron pans. For the production of a 60 per cent. caustic soda, the lye is evaporated until it shows 37° to 38° B., and a temperature of 281° F. The fire is then withdrawn and the contents of the pan allowed to rest for clarification and the deposit of the salts. For the production of 70 per cent. caustic soda the lye is concentrated in English factories to about 42° to 44° B. After allowing the contents of the pan to settle, the clear lye is drawn off into clarifying tanks and the remaining salt brought by a perforated scoop into a filter to be returned, after draining off, to the soda furnace. The clear lye is brought into an iron kettle, generally large enough to hold ten tons of caustic soda, and the fire is started. With a temperature of from 399° to 420° F., a reddish or sometimes a blackish froth forms on the surface. In most factories manufacturing 60 per cent. caustic soda this froth is, as a rule, removed; it is, however, absolutely necessary to remove it for 70 per cent. caustic soda. For caustic soda of less concentration it is advisable to interrupt firing when the temperature has reached 320° F., so that, by allowing the contents of the pan to clarify once more, more salts may separate and a purer

lye be obtained. Boiling is then continued. When the liquor has reached a temperature of 356° F., it already contains 53 per cent. of sodium oxide (nearly equal to 70 per cent. of caustic soda) and hardens completely on cooling. It is now very dark and of a syrupy consistency, and shows a great tendency to boil over. This is prevented by the workman beating the surface of the fluid in a peculiar manner with a shovel, which separates the froth. When the mass has reached a temperature of 401° F. boiling ceases almost entirely and but little vapor escapes, though the mass still contains nearly 20 per cent. of water. At about 460° F. the mass contains almost exactly 60 per cent. of sodium oxide = 77½ per cent. of caustic soda.

At this stage the contents of the pan show scarcely any motion, and a gentle bubbling is only noticed on the edge of the pan. Small quantities of caustic soda are carried along by the vapor which is evolved and cause a very unpleasant, stinging sensation upon the skin. The surface of the mass becomes coated with a lustrous froth of graphite, while reddish salts separate around the edge of the kettle. An iron lid is now placed upon the kettle and a hot fire kept up. When the mass has acquired the proper temperature, the oxidation of sodium sulphide still present, as well as that of the lower oxidation-degrees of sulphur, is completed either by the addition of sodium nitrate or recently by blowing in air. A sample is then taken and tested as to its alkalinity. The color of the mass varies from light brown to deep red. The contents of the pan are now allowed to clarify in the pan itself, which generally requires eight to twelve hours, a strong fire being kept up during this time. The quality of the product depends on complete clarification. The mass is then run into sheet-iron drums; if it is not entirely transparent and colorless the solid caustic soda will also show defects. The sediment in the pan, which amounts to about 9 to 11 per cent. of the mass, is generally brought into iron boxes and, after cooling, is broken up and again dissolved. The solution is brought to

28° B., allowed to settle, and the clear liquor added to the crude lye to be rendered caustic. The residue, which chiefly consists of ferric oxide, is thrown away.

For the production of 76 per cent. caustic soda it is preferable to remove the sodium sulphide from the diluted lyes, instead of waiting to the end of the operation. This is best effected by a suitable metallic oxide. The Greenbank Alkali Works Co., that first introduced caustic soda of a high degree, and furnish at the present time an excellent product, use plumbic oxide (or litharge) for the purpose.

*Commercial grading of soda.* The commercial grading of soda varies in the principal countries where it is produced. In Germany it is graded according to per cents. of sodium carbonate, in England according to per cents. of actual or available soda, and in France according to the test of Descroizilles founded upon an arbitrary basis. The grading according to German degrees seems to be the most rational one for ordinary soda, but is a very unfortunate one when applied to caustic soda. The English method, according to per cents. of available soda, is decidedly the best. Under available soda is understood everything that acts upon the test acid, because in the manufacture of soap it acts in the same manner as soda. In France these degrees are called Gay-Lussac's degrees; they are, however, never used in practice. Pure sodium carbonate contains, according to the English designation, 58.49 per cent. As sodium oxide is understood as a constituent of the hydrate, it is perfectly correct to apply the same degrees to caustic soda. If an Englishman, therefore, speaks of a soda of 52 per cent., it means only that the test acid neutralized by the soda corresponds to a quantity of  $\frac{52}{58.49}$  of the weight of sodium oxide used, which may, however, be present as carbonate, silicate, aluminate, hydrate, and even as sulphate. Unfortunately, in practice an error has crept into this otherwise rational designation which thus far has only been partially eradicated. The old incorrect equivalent of sodium of 32 is still used instead of 31, and the test acids have been regulated accord-

ingly. Thus too high a percentage is obtained, which does not correspond to the actual percentage of sodium oxide. Moreover, there is a further difference between the Tyne and Lancashire manufacturers, the former basing their grading on the equivalent of sodium carbonate which is taken at 54 instead of 53, the test acid being prepared so that 1 liter of it saturates 54 grammes of pure sodium carbonate. Pure sodium carbonate, with this test acid, therefore, indicates  $\frac{32 \times 100}{54} = 59.26$

= 59.26 per cent., instead of 58.49 per cent, and hence 0.77 per cent. too much. Every English statement of degree shows, therefore, 1.316 per cent. too much of its total amount; 50 per cent. of actual sodium oxide shows, for instance,  $50 + 50 \times 0.0136 = 50.66$  English degrees, as calculated by the Tyne manufacturers, as well as by the principal commercial analysts in England. In Liverpool, however, a practice has been gradually established which is not even based upon an erroneous equivalent, but simply intended to deceive the consumer. It is there customary to say: "As the 'old' equivalent of the pure sodium carbonate is  $\frac{1}{4}$  larger than the 'new,' all we need to do is to increase our per cent. figures found according to the actual equivalent by  $\frac{1}{4}$  in order to obtain the commercial grading; hence we call 53 per cent. sodium oxide 54 per cent." It may, however, be stated that certain large English firms send out their soda ash according to the real equivalent.

The most irrational of all methods of grading is that according to degrees of Deseroizilles, in general use in France. These degrees indicate how many parts of pure mono-hydrated sulphuric acid are neutralized by 100 parts of the soda ash in question. As the equivalents of sodium carbonate and double oil of vitriol are as 53 : 49, 100 parts of pure sodium carbonate must require 92.41 parts of mono-hydrated sulphuric acid, and hence, show as many degrees of Deseroizilles.

To avoid the tedious calculation of one method of grading into the other, a comparative table is here given which shows

the actual per cents. of sodium oxide (Gay-Lussac's degrees), of sodium carbonate (according to German and English degrees), and Descroizilles's degrees.

Gay-Lussac's degrees.	German degrees.	English degrees.	Descroizilles's degrees.	Gay-Lussac's degrees.	German degrees.	English degrees.	Descroizilles's degrees.
30	51.29	30.39	47.42	54	92.32	54.71	85.35
31	53.00	31.41	49.00	55	94.03	55.72	86.93
32	54.71	32.42	50.58	56	95.74	56.74	88.52
33	56.42	33.43	52.16	57	97.45	57.75	90.10
34	58.13	34.44	53.74	58	99.16	58.76	91.68
35	59.84	35.46	55.32	59	100.87	59.77	93.26
36	61.55	36.47	56.90	60	102.58	60.79	94.84
37	63.26	37.48	58.48	61	104.30	61.80	96.42
38	64.97	38.50	60.06	62	106.01	62.82	98.00
39	66.68	39.51	61.64	63	107.72	63.83	99.58
40	68.39	40.52	63.22	64	109.43	64.84	101.16
41	70.10	41.54	64.81	65	111.14	65.85	102.74
42	71.81	42.55	66.39	66	112.85	66.87	103.32
43	73.52	43.57	67.97	67	114.56	67.88	105.90
44	75.23	44.58	69.55	68	116.27	68.89	107.48
45	76.94	45.59	71.13	69	117.98	69.91	109.06
46	78.66	46.60	72.71	70	119.69	70.92	110.64
47	80.37	47.62	74.29	71	121.39	71.93	112.23
48	82.07	48.63	75.87	72	123.10	72.95	113.81
49	83.78	49.64	77.45	73	124.81	73.96	115.39
50	85.48	50.66	79.03	74	126.52	74.97	116.97
51	87.19	51.67	80.61	75	128.23	75.99	118.55
52	88.90	52.68	82.19	76	129.94	77.00	120.13
53	90.61	53.70	83.77	77	131.65	78.01	121.71

#### POTASH.

Commercial potash forms a mixture of salts, the principal constituent of which is potassium carbonate. When calcined it is a hard, but light, porous, granular mass of a white color shading into pearl-gray, yellowish, or bluish. Separate pieces frequently show blue or red stains upon the fracture. The red coloration is due to ferric oxide, the gray to admixed particles of coal, and the blue to the formation of a small quantity of potassium manganate by the action of the alkali upon manganic oxide. Potash has a strong alkaline taste and is odorless. It readily dissolves in water, a considerable quantity of indissoluble constituents frequently remaining behind.

It absorbs moisture from the air, deliquescing thereby. Its solution shows an alkaline reaction. It melts at the beginning of a red heat.

Four varieties of potash are found in commerce: 1, from wood ashes; 2, from the residue of beet-root molasses; 3, from wool sweat (suint); 4, from potassium sulphate according to Leblanc's process.

*Potash from wood-ashes.*—The industrial manufacture of potash from wood-ashes is carried on only in countries where wood is abundant, as in Russia, Illyria, Hungary, and the United States.

The process of manufacture is very simple and may be divided into five operations: 1. Incineration of the plants; 2. Lixiviation of the ashes; 3. Evaporation of the lye; 4. Calcination of the crude potash; 5. Purification of the potash.

Little need be said about the *incineration* of the plants. The slower the combustion, however, the more ash is obtained. With a vigorous combustion not only a considerable portion of the ash is carried off by the draught, but a portion of the alkaline salts evaporates on account of the high temperature.

The object of *lixiviating, leaching, or washing the ash* is to separate the soluble salts from the insoluble. The former consist chiefly of potassium carbonate, sulphate and chloride. It is *not* indifferent whether leaching is effected with cold or hot water. Potassium carbonate and chloride dissolve readily in cold water, potassium sulphate, however, with difficulty. By leaching, therefore, with cold water less potash is obtained, but it is richer in potassium carbonate. But potassium sulphate, being also a very valuable salt, leaching is generally effected with hot water.

Lixiviation is mostly carried on in wooden vats about  $3\frac{1}{4}$  feet high and  $3\frac{1}{4}$  to 5 feet in diameter. They are provided about 4 to 6 inches above the actual bottom with a perforated bottom, in which is inserted a vertical pipe for the escape of air expelled by the water, which otherwise would have to force its way through the ash. To prevent insoluble constituents of

the ash from being carried away, the perforated bottom is covered with a straw mat, or with a layer of straw several inches thick, over which is generally placed a piece of coarse linen. On one side, immediately below the perforated bottom, the vat is provided with a discharge-cock. The space between the two bottoms becomes gradually filled with fine particles of ash passing through the straw, and must, therefore, from time to time be cleansed.

The ash is first moistened with water, then brought in small portions into the vat and rammed down. Lixiviation is carried on in a systematic manner, *i. e.*, several vats are placed together, forming what is termed "a battery." The lye obtained is of a brown color, due to organic substances extracted from the incompletely carbonized wood by the potassium carbonate. The residue from leaching ashes affords a valuable manure.

The *evaporation of the lyes* is mostly conducted in shallow cast-iron kettles. Alongside these kettles are placed sheet-iron pans which are heated by the fire under the kettles and serve for preliminary warming. The kettles and pans being filled, a strong fire is started. The evaporated liquid is constantly replaced by fresh liquor from the pans. When the liquor has acquired a syrupy consistency, the supply of fresh liquor from the pans is interrupted and the fire moderated. On the side of the kettle a salt crust separates, which becomes thicker and thicker until all the liquor is converted into a dry salt-cake, when firing is entirely discontinued. After sufficient cooling the brown, hard salt-cake is broken out by means of a hammer and chisel. This crude product, containing about six per cent. of water, is known in the trade as crude or lump potash. As this method of boiling down and cutting out the crude potash must evidently cause considerable damage to the iron pans, the operation is, in some instances, conducted in a somewhat different manner. The liquid is stirred with iron rakes, and the salt, instead of forming a hard, solid mass, is obtained as a granular powder containing upwards of 12 per cent. of water.

A special kind of potash, consisting chiefly of caustic potash with varying quantities of potassium carbonate, is produced in this country, and brought into commerce under the name of "Red American Potash" or "stone ash." For its production the crude potash liquor is heated to boiling, and a quantity of milk of lime added according to the intended degree of causticity. The carbonate of lime formed is allowed to settle, when the clear lye is drawn off and evaporated until no more water escapes. It is finally heated nearly to redness in the evaporating pan itself, or in a special pan of thick cast iron, in order to destroy the organic substances and to melt the potash. The liquid potash is then ladled out with a sheet-iron ladle into cast-iron boxes to congeal to a stone-hard mass permeated with bubbles. The product, after removal from the boxes, is broken up and immediately packed into tight barrels. It is always contaminated with ferrie oxide and has a dirty red or brown color.

The object of *calcining* the crude potash, which is effected in a reverberatory furnace, is to remove the last particles of water, and specially to destroy the organic substances to which is due the brown color.

Potash obtained from wood-ashes is, as previously mentioned, chiefly a mixture of potassium carbonate, sulphate and chloride. For some applications of potash these three are almost of equal value, as, for instance, for the fabrication of alum; for most purposes, however, the content of potassium carbonate is the valuable portion. Whether the article is poor or rich in the latter can be recognized by the behavior of potash on exposure to the air in an open vessel. Potassium carbonate is a very deliquescent salt, absorbing moisture from the air with avidity; if, therefore, potash rapidly becomes moist on exposure to air, it is an indication of its being rich in potassium carbonate.

*Potash from the carbonized residue of beet-root molasses.*—For many years wood-ash was the only source of potash. But of late years a source of potash has been found in the residue from the distillation of beet-sugar molasses.

The residue, in case it contains free acid, is first neutralized by an addition of calcium carbonate. It is then evaporated to dryness and the residue heated until carbonization of the organic substances is effected.

The composition of the carbonized residue varies, but may be gleaned from the following approximate analysis:

Potassium carbonate . . . . .	30 to 35 per cent.
Sodium carbonate . . . . .	18 to 20 "
Potassium chloride. . . . .	18 to 22 "
Potassium sulphate. . . . .	6 to 8 "
Insoluble matter . . . . .	28 to 15 "

In the insoluble matter are contained coal, calcium carbonate and phosphate.

While the carbonized residue is mostly manufactured in the molasses distilleries it is generally refined in chemical factories. The process is very simple, it being based upon the different proportions of solubility of the various salts. In many factories the carbonized residue was formerly simply lixiviated with water, and the liquor thus obtained evaporated to dryness, 100 pounds of carbonized residue yielding by this process 45 to 60 pounds of potash, which showed 50 to 60 alkalimetric degrees. Much of this kind of potash was brought into commerce during the Crimean war, when there was a want of Russian potash, and contributed much to the disfavor with which the article was considered by soap-boilers.

At the present time a separation of the separate salts is generally effected whereby a potash is obtained containing still 8 to 15 per cent. of sodium carbonate, which, however, can be reduced to 4 per cent. by redissolving and re-evaporating.

*Potash from wool sweat (suint).*—The wool of sheep is saturated with a yellow, fatty substance, the so-called sweat or suint, secreted by the skin of the animal. It consists of a combination of potassium with the nitrogenous acid of a special fat, which is partly in a free state and partly combined with earths to an insoluble soap, and of small quantities of

potassium carbonate and potassium acetate, alkaline chlorides and an odoriferous substance. Its content of sodium is small.

In order to reduce the consumption of fuel to a minimum it is necessary in working suint to obtain as concentrated liquors as possible. For this purpose the wool is pressed firmly into vats and lixiviated with water in such a manner that all the water passes through several lots of wool and that each separate lot of it is several times washed, finally with fresh water. The suint solutions are then evaporated to dryness, and the residue obtained thereby is heated to a red heat in iron retorts. During this operation a large volume of carburetted hydrogen gas and ammonia is evolved, which is passed through a purifying apparatus to eliminate the ammonia and to render the gas fit for illuminating purposes. The carbonized residue in the retort contains the alkaline salts which are obtained by lixiviation with water. The liquor thus obtained contains carbonate, chloride and sulphate of potassium, which are separated from each other by evaporating the liquor and crystallization.

*Potash from potassium sulphate.*—While the previously mentioned sources of potash are limited, its manufacture from potassium sulphate promises to become a branch of industry of unlimited extent. Much of the potash salts, which find their way into the English market, are derived from the so-called "Strassfurt salts," produced from the alkaline minerals found in enormous quantities in the valley of the Bode about twenty-five miles southwest of Magdeburg. The manufacture of potash from potassium sulphate was introduced into Germany, in 1861, by Vorster & Gruneberg, of Kalk, near Cologne. The manufacture of potassium chloride is based on the decomposition of carnallite contained in the raw material, in a hot saturated solution, potassium chloride crystallizing out, and magnesium chloride remaining in solution. The hot solution is brought to  $36\frac{1}{2}$ ° B., diluted to 35° B., run into settling tanks, and allowed to crystallize; these crystals, once refined, are almost pure potassium chloride. The next stages

of the process are almost identical with those of the Leblanc soda process, the raw material (potassium sulphate) being obtained either from potassium chloride and sulphuric acid by the ordinary sulphate process, or by decomposing the former with magnesium sulphate or kieserite. Equal weights of potassium sulphate and finely divided limestone or chalk, together with varying quantities of small coal, are roasted together in a reverberatory furnace, the product being an exceedingly impure potassium carbonate. When decomposition is complete, the molten mass is raked out, broken up when cool, and lixiviated in tanks. The soluble potassium and soda salts are thereby dissolved out and evaporated, and then calcined in a small reverberatory furnace.

A carbonate of better quality is produced by following more closely the carbonating operation of the soda process. The liquors from the tanks are evaporated, the potassium chloride and sulphate, which separate out during the concentration, being skimmed off, and sawdust is thrown in. The dried salts are then removed to the carbonator and exposed, at first, to a gentle heat, which is, however, finally urged to redness. By this process the sulphur compounds are oxidized into sulphate, and the caustic potash is converted into carbonate. The chief object of the sawdust is to keep the mass of salt open.

*Caustic potash.*—This article has for a number of years been brought into commerce by the Greenbank Alkali Works Company of St. Helens, England, and its manufacture is also to some extent carried on in Germany and other countries. The product, however, has the drawback of being too expensive, and as the lye prepared by the soap-boiler himself comes much cheaper, it is seldom used except perhaps for the purpose of increasing the strength of tank lyes. Caustic potash and caustic-potash lye are now also produced by the electrolytic process, the lye having a density of 50° B. and contains 48.4 per cent. potassium hydrate and 1.4 per cent. potassium carbonate. The caustic potash contains 9.0 per cent. potassium hydrate.

## CHAPTER VII.

### TESTING OF SODA AND POTASH.

THE value of the different varieties of soda and potash depends on their content of pure, effective substance, *i. e.*, of alkaline carbonate or caustic alkali. From the preceding chapter it will be readily understood that the quantity of effective substance varies very much in the alkalies found in commerce, and that the value of a particular kind of soda or potash can, therefore, be ascertained only by determining its content of pure alkali.

For the determination of the value of soda ash or soda crystals, it is sufficient to ascertain the sodium carbonate by the alkalimetric method. It is, however, different with caustic soda, where it is above all required to know how much caustic soda it contains. By directly testing caustic soda by the alkalimetric method an erroneous result is obtained, since the larger or smaller quantity of sodium carbonate always contained in it is also determined as caustic soda. To avoid this error the sodium carbonate has to be separated previously to titration, which is readily effected by means of barium chloride. By adding to caustic soda solution barium chloride solution a white precipitate of barium carbonate or sulphate is formed in the presence of sodium carbonate or sulphate, while no effect whatever is produced upon the caustic soda. For the examination of soda in this manner, dissolve a weighed quantity of it in hot water and add barium chloride solution until no more precipitate is formed. When all the precipitate has settled on the bottom, filter and rinse with hot water until the water running from the funnel shows no alkaline reaction. The caustic soda in the filtrate is then determined by the alkalimetric method.

With potash a correct result is never obtained by a me alkalimetric determination, since the sodium carbonate, which is never wanting even in the best potash, is then also calculated as potassium carbonate. The error resulting in this manner is the more serious, as sodium carbonate has a lower atomic weight than potassium carbonate and consequently requires a larger quantity of acid for saturation. If, therefore, with a potash containing soda, the alkalimetric degrees four are calculated as potassium carbonate, a larger sum total than the quantity of the two alkaline carbonates together amounts to is obtained. Thus, for instance, a potash which contains 8 per cent. potassium carbonate and 5 per cent. sodium carbonate shows an alkalimetric content of 91.62 per cent., and potash with 85 per cent. potassium carbonate and 8.6 per cent. sodium carbonate, one of 96.22 per cent. A thorough analysis, therefore, is required for the determination of the value of potash, and as this requires considerable experience in such work, it is best to employ a skilled chemist.

*Alkalimetry.*—The testing of soda and potash by the alkalimetric method is such a simple process that it can be readily learned without much preliminary knowledge of chemistry. To make the processes taking place in the execution of the test clear to persons without chemical knowledge, it will be necessary to mention a few chemical laws.

It is a fact determined by innumerable experiments and confirmed by daily experience, that by two bodies acting upon each other, a third body, or, as it is called, a chemical compound, is formed which exhibits properties entirely different from those of either of its constituents, and the proportions of weight of these constituents which form that particular compound admits of no variation whatever. By long-continued trituration, for instance, of mercury and sulphur, cinnabar is formed, the two bodies, however, combining only in the proportion of 200 parts by weight of mercury and 32 parts by weight of sulphur. Every excess of mercury or sulphur above this proportion fails to form cinnabar, the excess of mercury

sulphur, as the case may be, remaining behind. In the same manner 23 parts by weight of sodium combine with 35.5 parts by weight of chlorine to common salt. The numbers which express the proportions of weight in which bodies form combinations are called atomic weights. By careful experiments chemists have determined the atomic weight of every body and have found that, for instance, the atomic weight of sodium is 23, that of potassium 39, that of calcium 40, that of sulphur 32, that of chlorine 35.5, that of carbon 12, that of oxygen 16, that of hydrogen 1, etc. If, therefore, two or three of the mentioned bodies form a combination, it is done in the proportions of the numbers given. To this must, however, be added that the bodies may also combine in the proportions of twice, three times, or four times the above numbers. Water, for instance, is a combination consisting of  $2 \times 1$  part by weight of hydrogen and 16 parts by weight of oxygen. Soda or sodium carbonate consists of three bodies, viz.,  $2 \times 23$  parts by weight of sodium, 12 parts by weight of carbon, and  $3 \times 16$  parts of oxygen. Hence the chemical law: *When two or more bodies combine chemically, it is done in the proportion of the atomic weights or their simple multiples.*

This most important of all chemical laws finds application in the determination of the constituents of various bodies. The branch of chemistry which occupies itself with the determination of the constituents of bodies is called "chemical analysis." There are two methods of determining the quantity of a body in a given substance; viz., gravimetric analysis, or analysis by weight, and volumetric analysis, or analysis by measure or process of titration (analysis by means of standard solutions). By the first method the substance to be examined is reduced to forms or combinations which are most accurately known as regards the proportions of the quantity of their constituents and admit at the same time of a sharp determination of weight. By volumetric analysis, on the other hand, the quantity of a body is found by reducing it from a determined condition into another also determined condition, by the assistance of a liquid

of known effective value and under conditions allowing of a plain recognition of the determination of the reduction. In alkalimetry this final point is recognized by a change of color.

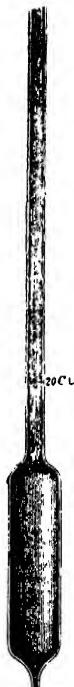
For volumetric determinations a few instruments are required which shall be briefly described as follows:—

For the preparation of the *test liquids* or *standard solutions* required for volumetric analysis, serves a flask, Fig. 6, which, up to a mark on the neck, holds exactly 1 liter or 1000 cubic centimeters, and can be closed with a glass stopper. There

Fig. 6



Fig. 7.



are also flasks of the same shape with a capacity of 500, 200, and 100 cubic centimeters. For measuring out small quantities of liquids serve a burette and a pipette, the latter a glass tube of the form as shown in Fig. 7. It is filled by dipping the lower end into the liquid and sucking on the upper with the mouth until the liquid has ascended nearly to the top. The upper end is then quickly closed with the index finger of the right hand. By slightly lifting the finger, the liquid is then allowed to flow off by drops until its level has reached a mark above the

convex expansion when it will contain exactly the number of cubic centimeters indicated opposite to the mark. There are pipettes of 50, 20, 10, 5, and 1 cubic centimeters capacity.

The burette is a cylindrical glass tube open on the top, graduated, commencing from the top, into whole, one-tenth and one-fifth cubic centimeters. The lower end of the tube is drawn out to a somewhat distended point so as to allow a

Fig. 8.

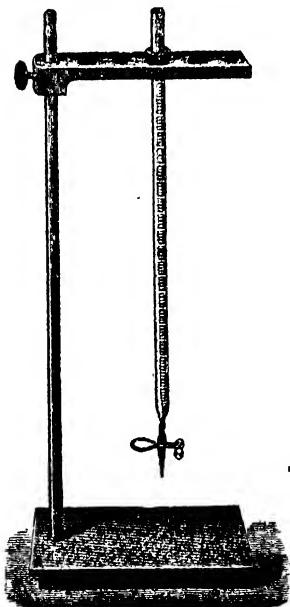
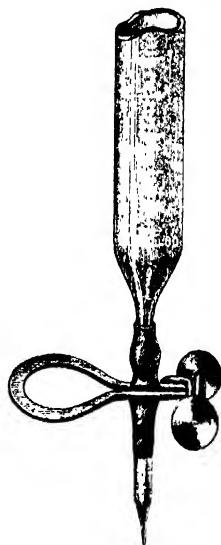


Fig. 9.



rubber tube to be drawn over it and securely fastened. In the lower end a glass tube drawn out to a fine point is inserted. The rubber tube is compressed in the centre by a clip or compression stop-cock, whereby the lower end is closed. Fig. 8 shows a burette secured in a stand, while Fig. 9 shows the lower part with the clip on a larger scale. The burette is

filled with liquid from above by means of a small funnel. By a quick, strong pressure upon the handle-joint of the clip, some liquid is then allowed to flow in a jet into a vessel. By this the tube below the clip is filled with liquid and the air contained in it expelled. By a slighter or stronger pressure the liquid can, after some experience, be ejected in drops or in a stronger jet. The number of cubic centimeters which have been allowed to flow out can be readily read off by keeping the surface of the liquid in the tube on a level with the eye.

The determination of effective substance in the alkalies executed with the assistance of the above instruments is based upon the following consideration : Caustic soda lye is a solution of caustic soda in water. By mixing some caustic soda lye with (dilute) sulphuric acid a chemical combination takes place, the result being sodium sulphate (Glauber's salt). Caustic soda and sulphuric acid combine, however, only in determined proportions of weight, namely, 40 parts by weight of pure caustic soda combine with 49 parts by weight of concentrated sulphuric acid. Every excess of caustic soda or sulphuric acid above this proportion remains caustic soda or sulphuric acid and is not converted into Glauber's salt. Now caustic soda, sulphuric acid, and Glauber's salt are representatives of three classes of chemical combinations known as bases or alkalies, acids, and salts. These three combinations can be distinguished from each other by the coloring substance known as litmus, a solution of which in water is termed litmus tincture. By coloring water blue with litmus tincture and adding one drop of sulphuric acid, the liquid turns red ; it, however, becomes blue again by adding caustic soda lye. But salts, such as Glauber's salt, do not change the color of either blue or red litmus tincture.

This action of the alkalies and acids upon litmus is utilized for our purposes. When to caustic soda solution sufficient litmus tincture be added to perceptibly color the liquid blue, and to this blue liquid, carefully and with constant stirring, sulphuric acid, until it just acquires a red coloration, then

exactly that quantity of sulphuric acid will have been added, which, with the caustic soda present, forms Glauber's salt, *i. e.*, caustic soda and sulphuric acid have been mixed in the proportion of 40 : 49 parts by weight.

Suppose, now, we had 1 liter of dilute sulphuric acid containing exactly 49 grammes of concentrated sulphuric acid and 1 liter of caustic soda lye, containing exactly 40 grammes of caustic soda (these solutions are known as standard acid solution and standard alkali solution), by pouring these two liquids together a liquid would be formed, which contains neither sulphuric acid nor caustic soda, but Glauber's salt, and as a proof that neither one of them is present, the color of neither red nor blue litmus tincture would be changed by an addition of any of the liquid. Likewise, the quantities of acid and alkali contained in 100, 10, or 1 cubic centimeters of the two solutions would be just sufficient for the formation of Glauber's salt. In place of litmus tincture for testing a substance as to whether it is an acid, or an alkali or a salt, litmus paper may be used. It is white unsized paper colored blue by dipping it into litmus tincture, and red by dipping it into the blue solution to which dilute hydrochloric acid has been carefully added until the color has just changed to red; an excess of acid should be avoided. For use the paper is cut up into strips. The salt solutions, at least those which come here into consideration, are so constituted as not to change the color of blue or red litmus paper; they show a neutral reaction.

From what has above been said, it follows that by adding to a large quantity of caustic soda solution 1 liter of dilute sulphuric acid containing 49 grammes of pure sulphuric acid, this acid combines with 40 grammes of caustic soda to form Glauber's salt, and, further, that by adding only  $\frac{1}{10}$ ,  $\frac{1}{100}$ , or  $\frac{1}{1000}$  liter, or 100, 10, or 1 cubic centimeters of acid,  $\frac{1}{10}$ ,  $\frac{1}{100}$ , or  $\frac{1}{1000}$  part of 40 grammes of caustic soda is converted into Glauber's salt. By adding, therefore, to a solution of caustic soda of unknown content of the above standard sulphuric acid until the added litmus tincture just turns red, the content of

caustic soda can be determined by the quantity of sulphuric acid used. If, for instance, 12.5 cubic centimeters of standard sulphuric acid had been used, and as one cubic centimeter of acid indicates  $\frac{1}{100}$  gramme or 0.040 gramme of caustic soda, the liquid would contain  $0.040 \times 12.5$  grammes = 0.5 gramme of caustic soda.

The preparation of the standard sulphuric acid would offer no difficulty if the so-called concentrated sulphuric acid found in commerce were pure sulphuric acid. It always contains, however, some water, though the quantity is so trifling that for our purposes, where the highest degree of exactness is not demanded, it may be considered as sufficiently pure. In every drug store can be found sulphuric acid of nearly 1.842 specific gravity, which is the specific gravity of pure acid. If, however, it cannot be obtained of this strength, it can be readily brought to the commercial standard by carefully adding fuming or Nordhausen sulphuric acid.

Weigh out of this acid, as accurately as possible, 49 grammes, pour it into the liter flask (Fig. 6), rinse the glass in which it has been weighed several times with water, and pour the latter into the liter flask. Then add sufficient water to fill the flask one-fourth full and shake. As by mixing the water and sulphuric acid the liquid becomes heated, allow it to stand for about one hour, and then fill the flask with water exactly to the mark on the neck. After shaking, close the flask with a glass stopper and preserve the solution for use.

As the standard acid solution prepared in the above manner is not exactly standard, and an error (at the utmost of  $\frac{1}{4}$  per cent.) may be made in the determination of soda, potash, etc., oxalic acid is used for the preparation of the standard acid solution in place of sulphuric acid, where the highest degree of accuracy is required. Pure oxalic acid can be readily prepared from the commercial article by dissolving  $\frac{1}{2}$  pound of it in as little hot water as possible, filtering the hot solution and allowing it to stand quietly in a cool place. In twenty-four hours the oxalic acid will have separated in acicular crystals,

which are taken from the water and spread out upon blotting-paper to dry, when they are ready for use.

Solution of oxalic acid behaves towards alkalies in the same manner as sulphuric acid, 40 grammes of caustic soda combining with 63 grammes of crystallized oxalic acid. Therefore, to prepare the solution, weigh out 63 grammes of crystallized oxalic acid, bring it into the liter flask and pour about  $\frac{3}{4}$  liter of water over it. After effecting the solution of the oxalic acid by shaking, fill the flask up to the mark on the neck with water and close it with a glass stopper.

Besides standard acid there is required a standard caustic soda solution containing 40 grammes of caustic soda in the liter. The preparation of this solution is somewhat more difficult, since the solid caustic soda of the shops is by no means pure, containing varying quantities of water. It is, therefore, necessary to choose a roundabout way in order to obtain a caustic soda solution with an accurately determined content of caustic soda. For this purpose dilute about 300 cubic centimeters of pure caustic soda lye with about 500 cubic centimeters of water. Of this liquid measure out into a glass, by means of one of the pipettes previously described, 10 centimeters, color with blue litmus tincture, and dilute with about 100 cubic centimeters of water. Then add, drop by drop, by means of the previously described clip burette, stirring constantly, standard acid, until the liquid acquires a distinctly red color, which indicates that exactly the quantity of acid required for the neutralization of the caustic soda present has been added. The cubic centimeters of acid used are read off on the burette. Now as 1 cubic centimeter of acid saturates exactly 0.040 gramme of caustic soda, the quantity of caustic soda contained in the 10 cubic centimeters of caustic soda solution can be readily found from the quantity of acid used by multiplying the number of cubic centimeters used by 0.040. Suppose 15.5 cubic centimeters of acid had been used, 10 cubic centimeters of the above caustic soda solution would contain as much caustic soda as is required for the saturation of the

acid in the 15.5 cubic centimeters of standard acid. Now, in order to prepare from this caustic soda solution a solution sufficiently diluted so as to become saturated cubic centimeter for cubic centimeter with the standard acid solution, 10 cubic centimeters of it must be sufficiently diluted to make 15.5 cubic centimeters, or as,  $10 : 15.5 = 645 : 1000$ , 645 cubic centimeters of soda lye have to be diluted to 1000 cubic centimeters. These 645 cubic centimeters are then measured out into the liter flask, and after adding water up to the mark on the neck, the flask is tightly stoppered and the contents are shaken.

After these general explanations the manner of testing the alkalies in regard to their effective substance, and, therefore, their real value to the soap-manufacturer, by means of the above-mentioned standard solutions, will be given.

For the determination of the effective substance in the alkalies, a determined quantity has to be weighed out; for soda and potash this is best done as follows: Cut out two square pieces of smooth writing paper of exactly the same size, and place one upon each dish of a balance. Upon one paper place the weight and upon the other bring the substance to be weighed in small pieces until the equilibrium is restored. Caustic soda and caustic potash can be weighed in the same manner, but they being very hygroscopic, which augments their weight, it must be done in a dry-room, and as quickly as possible. It is best, however, to weigh them inclosed in small glass-tubes tightly stoppered.

Next dissolve the quantity weighed out in about 100 to 150 cubic centimeters of water, color the solution blue with some litmus tincture, then add by means of the clip burette standard acid until the liquid acquires a distinctly red color, and read off the quantity of acid used.

For the examination of caustic soda it is best to weigh out 4 grammes and proceed as above. As each cubic centimeter of standard acid used indicates 0.04 gramme of caustic soda in the substance under examination, each cubic centimeter used, calculated to per cents., indicates 1 per cent. caustic soda.

To draw a simple conclusion as regards the per cent. content of a caustic potash from the number of cubic centimeters of acid used, it is best to weigh out 5.6 grammes, each cubic centimeter used indicating then 1 per cent. of caustic potash. For the determination of caustic soda or caustic potash in a soda lye, weigh out of the former in a small beaker 0.4 grammie and of the latter 5.6 grammes, pour the lye into a larger beaker, dilute with 100 to 150 cubic centimeters of water, add litmus tincture, and then standard acid until the liquid acquires a distinctly red color. For each cubic centimeter of standard acid 1 per cent. of effective substance is then to be allowed for in the calculation.

Namely, as 40 grammes of caustic soda and 56 grammes of caustic potash combine with 49 grammes of sulphuric acid and 63 grammes of oxalic acid, each cubic centimeter of standard acid indicates 0.04 grammie of caustic soda and 0.056 grammie of caustic potash; 100 cubic centimeters, therefore, respectively 4.0 and 5.6 grammes. If, now, in 4.0 grammes of solution or solid substance used are 4.0 grammes of caustic soda, it would indicate a pure substance or 100 per cent. Hence it follows that if, for example, only 15 or 65 cubic centimeters of standard acid are used, only 15, or 65 per cent. of pure substance is contained in the sample under examination, *i. e.* the number of cubic centimeters used gives at once the content in per cents.

For the determination of the value of soda or potash the process varies somewhat from the above. By adding to a solution of alkaline carbonate litmus tincture, the liquid acquires a blue color. By adding acid, carbonic acid escapes, which, however, remains partly dissolved in the liquid and colors the litmus tincture red. The quantity of acid required for the decomposition of the alkaline carbonate, therefore, cannot be recognized by the change of color. The object is, however, attained as follows: Add sufficient acid, so that the liquid shows a deep red coloration, and boil, whereby the carbonic acid is entirely expelled. If after a few minutes' boiling the

liquid still remains red, an excess of acid has been added. Now the standard soda solution, prepared as previously described, is so constituted that 1 cubic centimeter of it exactly neutralizes 1 cubic centimeter of acid ; by adding, therefore, to the liquid freed from carbonic acid by boiling, standard soda solution until the red color just passes into blue, exactly as many cubic centimeters of it will have been used as cubic centimeters of acid had been added in excess. By deducting these cubic centimeters of standard soda solution from the cubic centimeters of standard acid originally used, the exact quantity of standard acid required for the saturation of the alkaline carbonates is obtained.

As 53 grammes of sodium carbonate combine with 49 grammes of pure sulphuric acid or 63 grammes of oxalic acid, it is best to weigh out 5.3 grammes of soda, place them in a small flask of about 200 cubic centimeters' capacity, pour in water, and then heat. When the soda is dissolved, color blue with litmus tincture and add, by means of the clip burette, standard acid until the liquid acquires a deep red color. After continuing the boiling for a few minutes, the liquid should still show a deep red coloration. Then add, by means of the clip burette, standard soda solution, drop by drop, until the liquid, after shaking, just acquires a blue color. Deduct the quantity of standard soda solution used from the quantity of standard acid solution added ; the number of cubic centimeters obtained gives the content of pure sodium carbonate in per cents.

For the examination of potash by volumetric analysis, weigh out 6.9 grammes, since 69 grammes of potassium carbonate are neutralized with 49 grammes of sulphuric acid, and proceed in the same manner as given for soda. Each cubic centimeter of standard acid used for neutralization indicates 1 per cent. of potassium carbonate.

To those not having a balance for accurately weighing 4, 5.3, and 6.9 grammes, it is recommended to weigh out larger quantities, namely, 40 grammes of caustic soda, 53 of calcined

or crystallized soda, and 69 of potash, and dissolve in sufficient water for the solution to make exactly 1 liter. For the execution of the test take of this solution, by means of a pipette, exactly 100 cubic centimeters, when each cubic centimeter of standard acid used will correspond to 1 per cent. of caustic soda or sodium carbonate or potassium carbonate.

Standard solutions, already prepared, can generally be had in almost all large drug stores at moderate prices.

Of the standard acid solutions—

1 cubic centimeter	0.047	grammes of potassium.
	- 0.056	" caustic potash.
	- 0.069	" potassium carbonate.
	= 0.031	" sodium.
	= 0.040	" caustic soda.
	- 0.053	" sodium carbonate.

Of the standard alkaline solutions—

1 cubic centimeter	- 0.064	grammes of oxalic acid.
	- 0.010	" sulphuric acid.
	- 0.049	" concentrated sulphuric acid.

In the directions given above litmus tincture has been prescribed as an indicator because it is best known. Its use for this purpose has, however, the drawback that, if the alkali solution compounded with standard acid solution contains carbonic acid, it has to be boiled until the latter is entirely expelled, and the addition of acid has to be continued until after continuous boiling the color remains pure red. The use of methyl-orange is more convenient and more rapid. Its solution in water is yellow, turning red when an acid is added. Carbonic acid does not act upon it, and titration can, and even should, be effected cold. Enough methyl-orange solution is added to the lye to produce a just perceptible pale yellow coloration; acid is then added, whilst shaking constantly, till the yellow color passes into a purple-red. The reverse change takes place with the same sharpness in titrating acids with free alkalies or even with alkaline carbonates. For titrating

alkali solutions with methyl-orange as an indicator sulphuric acid has to be used, oxalic acid not being available for this purpose.

Phenolphthalein is an excellent indicator for solutions which only contain caustic alkalies. If an alcoholic solution of it be added to a fluid having an acid reaction, it remains colorless; if, however, the smallest quantity of alkali be added to phenolphthalein solution, the fluid at once acquires an intense rose color. The change to red is so sudden as to leave no room for deception as is possible with the gradual change of the litmus tincture from red to blue. However, phenolphthalein has the same drawback as litmus, that the red color does not appear in the presence of carbonic acid and the latter has to be expelled by boiling. It must also be mentioned that ammonia does not produce a red coloration in phenolphthalein solution.

*Taking a sample for analysis.*—For the examination of soda or potash, or of any other commercial article, it is necessary to procure an average sample. For this purpose take from every barrel or drum a sample weighing about three-quarters to one pound and put it into a barrel. The latter should be tightly covered to prevent the original composition of the samples from suffering a change by the evaporation or absorption of moisture.

When a sample has been taken from all the barrels or drums, turn the barrel containing them over on a level, hard, clean surface, and mix the mass by spreading it out uniformly in a shallow layer and reforming it into a pile in the center, carefully repeating the operation several times. From the mixture thus finally obtained make up a sample by combining small portions from every part of the mass spread out over the surface. This sample is then again manipulated in the same manner as the original large sample, and then distributed into several bottles tightly corked and sealed. This distribution into several bottles is advisable in order to have evidence in case of dispute. Taking the samples and all other operations

connected with it must of course be effected as rapidly as possible to avoid the danger of the sample suffering a change by the absorption of moisture or by evaporation. For actual use the samples have to be further pulverized and mixed.

When taking the first average sample of substances consisting of a mixture of large and small pieces, care must be taken to preserve the same proportions as in the original. After mixing the various samples, crush the large pieces, again mix the whole, and finally take a small sample from every portion of the mass. This sample is pulverized and preserved as above described.

Where there is a large number of barrels, so that the taking of a sample from each barrel would be tedious and time-consuming, a sample is taken from every third, fourth, fifth, or tenth barrel, according to the number, and the samples mixed in the previously described manner. For taking samples from barrels it is best to use the instrument shown in Fig. 10. After boring by means of a center-bit a hole about twelve inches wide in the head of the barrel, the instrument is forced with a screw-like motion as far as possible into the barrel, whereby the concavity becomes filled with portions of the various layers of the contents. The instrument is then withdrawn without turning. It should be always kept bright and free from rust.

Sample-taking, as previously mentioned, should be done as quickly as possible. Samples of potash and similar substances, which are hygroscopic, but do not attack the human skin in the same degree as caustic alkalies, are best taken by pushing the hand as deeply as possible into the barrel.

In taking samples of caustic alkalies care must be chiefly had to break up large pieces and take portions from the center, or to scrape off the external crust and use the inner portions. This is necessary, because the surfaces of caustic alkalies generally undergo considerable changes by the absorption of moisture and carbonic acid and their consequent conversion

FIG. 10.



into carbonates. It must further be taken into consideration that the alkalies do not always show the same content in all portions of the drums. This is due to the fact that the foreign salts, etc., which contaminate the product are not separated uniformly with the congealing alkali, but remain longer in solution and accumulate in the central portion of the drum which congeals last. The best average sample is, therefore, obtained by taking pieces from the bottom and sides of the drum.

## CHAPTER VIII.

### AUXILIARY RAW MATERIALS.

*Water*.—Water ( $H_2O$ ) plays a prominent and indispensable part in the manufacture of soap, and may well be called a raw material. In a pure state it is tasteless and inodorous, and colorless in small bulk, but has a greenish or blue color when viewed in bulk. It is an exceedingly bad conductor of electricity of low electromotive force. It attains its greatest density towards  $40^\circ$  F., freezes at  $32^\circ$  F., expanding thereby by  $\frac{1}{11}$  of its volume, and forms in this respect an exception to the general law that bodies contract, *i. e.*, decrease in volume, the more they are cooled. It boils under the ordinary pressure at or near  $212^\circ$  F. It evaporates at all temperatures.

Water is a solvent for bodies of the most varying nature. The quantities of such bodies which dissolve are, as a rule, dependent at the temperature at which solution takes place, more being generally dissolved hot than cold. A solution saturated when hot, *i. e.*, one which at this temperature is incapable of taking up more, again deposits, on cooling, a portion of the dissolved substance, and is then called *cold saturated*. Some substances are to such extent soluble in water that they even withdraw aqueous vapor from the atmosphere in order to dissolve in it. Such substances liquefy in the open air, and are called *deliquescent*. On the other hand, some substances containing water yield the latter to air not saturated with moisture and, while originally crystallized, decompose to powder. They are called *efflorescent*.

Gases are also soluble in water or, as it is generally termed, are absorbed by it. The solubility of gases, however, varies very much and is dependent on the temperature and the pres-

sure upon the water. The solubility of a gas in water is the greater, the lower the temperature of the water, and the greater the pressure exerted upon the solution by the gas not absorbed.

Water as found in nature always contains partly in a finely divided, or in a dissolved state, varying quantities of substances with which it comes in contact. Rain-water, the purest form of natural water, contains traces of ammoniacal salts and other impurities which it collects from the atmosphere during its descent. So soon as it reaches the earth it begins to dissolve small portions of various solid materials with which it comes in contact. The waters of wells, springs and rivers differ very much from each other, according to the nature of the layers of rock and earth over or through which they have passed, and from which they dissolve a great variety of substances, such as sodium sulphide and chloride, magnesium sulphate and carbonate, calcium sulphate and carbonate, carbonic acid, silica, etc.

Of the above constituents held in solution by water the carbonates and sulphates of lime and magnesia are the most injurious ones for most purposes.

Household experience has established a classification of the waters from natural sources into *soft* and *hard* waters, a division which depends chiefly upon the manner in which they act upon soap. Hard water yields a lather with soap only after the whole of the calcium and magnesium salts have been thrown down from the water in the form of an insoluble lime-soap. This, of course, causes a considerable loss in large establishments where a great deal of soap is used, since the lime-soaps formed are insoluble and act neither mechanically nor chemically.

This peculiar property which water acquires by the presence of calcium and other salts is termed *hardness*. The simplest means to determine the hardness of a natural water is by a solution of 1 part of pure fatty acid soap in 100 parts of highly rectified alcohol, and adding 150 parts of water. By adding

some of this reagent to distilled water the mixture remains clear, and on shaking yields a lather which stands four or five minutes. If the water, however, contains lime or magnesia, or both, a turbidity caused by calcium or magnesium oleate appears, which is the greater the larger the amount of these mineral substances in the water; but with very hard water caseous flakes are formed. On shaking, no permanent lather is formed until sufficient of the above-mentioned soap solution has been added to decompose the whole of the calcium and magnesium salts, and the soap solution somewhat predominates.

A simple remedy for hard water is a solution of silicate of soda of 20° B., 5 per cent. of which added to the water will cause the lime, etc. to precipitate and leave the water sufficiently pure for use. In the preparation of alkaline lyes this is effected spontaneously as the free carbonic acid in the lime is precipitated on causticizing with lime.

For technical purposes turbid and impure water can be purified by allowing it to stand and by subsequent filtration through sand. Substances dissolved in the water are, however, not removed by filtration. Perfectly pure water can only be obtained by distillation, *i. e.* by heating the water to boiling and again condensing in a suitable apparatus the steam to water.

In the manufacture of soap the constitution of the water exerts no material influence. It chiefly serves for dissolving the alkalies. If caustic alkali is dissolved in water containing carbonic acid, an equivalent quantity of the alkali is converted into alkaline carbonate and hence is no longer capable of saponifying neutral fats. If water containing carbonic acid is used in the preparation of lye, an equivalent quantity of caustic alkali is converted into carbonate of lime, becoming thereby ineffective. This is, however, of little importance, since an excess of lime is always added. If water containing gypsum be employed for the preparation of lye, the gypsum (sulphate of lime) is converted with the alkaline carbonates,

carbonate of lime and alkaline sulphate being formed, which are ineffective for the saponification process.

*Lime.*—Burnt lime or quick lime ( $\text{CaO}$ ) is the monoxide of the metal calcium. Neither the metal itself nor its oxide occurs as such in nature, though compounds of the metal with acids are very abundant and are widely distributed. Among them may be enumerated the carbonate, which exists as chalk, limestone, marble, cale-spar, etc., the sulphate as gypsum and selenite, the fluoride as fluor-spar, and the phosphate as apatite and bone-earth.

Water containing carbonic acid coming in the interior of the earth in contact with lime salts, becomes hard water by the absorption of calcium carbonate and sulphate. From the water the lime combinations reach the plants, and from them and the water the animal organism. The ash of plants contains calcium carbonate, phosphate and sulphate; the shells of eggs, oysters, etc., consist almost entirely of calcium carbonate; and bones contain about half their weight of calcium phosphate and carbonate.

By heating calcium carbonate ( $\text{CaCO}_3$ ) to from  $1140^\circ$  to  $1482^\circ$  F. the carbonic acid is expelled, while calcium monoxide remains behind. Upon this is based the process of lime-burning. With too high a temperature the small quantity of silica and alumina contained in the limestone becomes sintered over the surface, which prevents the escape of the carbonic acid; such lime is called *overburnt*. Notwithstanding the considerable loss of weight (over 40 per cent.) the calcium carbonate suffers by burning, its bulk does not decrease to any extent, the burnt lime remaining behind as a porous mass. The freer the limestone from foreign admixtures the better the lime produced from it. When the limestone consists chiefly of pure carbonate of lime, the resulting product is, what is termed, "*fat lime*." On the other hand, if the limestone contains magnesium carbonate (dolomite  $\text{CaCO}_3\text{MgCO}_3$ ) the resulting lime forms a short, thin pulp with water and is termed "*poor*." The latter cannot be recommended for the preparation of lyes.

Pure lime is white and often of considerable hardness; the ordinary commercial article, however, has generally a grayish-yellow color and contains some alumina, ferric oxide, etc. It rapidly combines with water under the development of considerable heat. If very little water is added it breaks up and decomposes to a soft, white powder, with a burning and caustic taste, which in bulk exceeds three times that of the lime used. This conversion is known as "slaking" and the product as "slaked lime" or calcium hydrate ( $\text{CaH}_2\text{O}_2$ ). During the operation of slaking so much heat is liberated that gun-powder may be ignited and wood carbonized.

Since by the absorption of moisture and carbonic acid from the air, lime becomes decomposed and ineffective, it has to be used while fresh, or be stored so as to exclude the air. It is advisable to slake it to a stiff paste and put it into a brick-lined pit, in which it will keep for months, carbonic acid being absorbed only by the upper layer, which comes in contact with the air. When no lime has for some time been taken from the pit, the upper layer should be removed, it being unfit for the preparation of lye.

In the manufacture of soap lime serves for the purpose of converting alkaline carbonates into caustic alkalies. Its value depends on its content of calcium oxide and may be determined as follows: Slake 10 grammes of the lime with water in a dish, pour a 10 per cent. sugar solution over it, rinse the whole into a one-half liter flask, fill up the flask to the one-half liter mark with a 10 per cent. sugar solution, and allow the whole to stand 24 hours with frequent shaking. Then pass the whole through a filter, take 50 cubic centimeters of the filtrate, color it blue with litmus tincture, and titrate with standard sulphuric acid until the liquid acquires a red coloration. By multiplying the cubic centimeters of acid used with  $0.028 \times 10$ , the per cents. of caustic lime contained in the sample are obtained. Good limestone should not contain less than 82 per cent. of calcium oxide, and, if possible, over 86 per cent.

*Common salt or sodium chloride* ( $\text{NaCl}$ ) is of great importance for all industries. It is the source of nearly all the soda used for technical purposes, of all the chlorine, hydrochloric acid, chloride of lime, and all products containing chlorine. It occurs as a mineral in beds of rock salt in widely-scattered districts, and in solution as brine in sea water and salt springs. It is obtained either by evaporation of its solutions (sea water and water of salt springs), or, where it occurs in extensive beds as rock salt, by mining. The latter is frequently red, blue or brown in color and is decolorized by dissolving and clarifying.

Perfectly pure common salt is not hygroscopic, but is made so by admixtures of magnesium and calcium chloride contained in the salt of commerce. Usually salt contains from 2.5 to 5.5 per cent. of water, not as a constituent but as an intermixture, hence the phenomena called decrepitation, due to the breaking up of the crystals by the action of the steam when salt is heated. Ignited to a strong red heat common salt fuses, forming an oily liquid, and at a white heat is volatilized without decomposition.

Common salt dissolves with almost equal ease in cold and hot water, a saturated solution containing nearly 27 per cent.

According to Poggiale, 100 parts of water dissolve of sodium chloride—

32.73 parts at . . . . .	5° F.	36.64 parts at . . . . .	104° F.
33.49 " . . . . .	14	37.25 "	140
34.22 " . . . . .	23	37.88 "	158
35.52 " . . . . .	32	38.22 "	176
35.63 " . . . . .	41	38.87 "	194
35.74 " . . . . .	48	39.61 "	212
35.87 " . . . . .	57	40.35 "	229.5
36.13 " . . . . .	77		

The following table shows the specific gravity of various solutions of common salt:—

## SODIUM CHLORIDE SOLUTIONS.

Specific gravity.	Per cents.						
1.00725	1	1.05851	8	1.11146	15	1.16755	22
1.01450	2	1.06593	9	1.11938	16	1.17580	23
1.02171	3	1.07335	10	1.12730	17	1.18404	24
1.028999	4	1.08097	11	1.13523	18	1.19228	25
1.03624	5	1.08859	12	1.14315	19	1.20098	26
1.04366	6	1.09622	13	1.15107	20		
1.05108	7	1.10384	14	1.25931	21		

1.20314 26.395 per cent.

In the manufacture of soap common salt is used for separating the soap from glycerin, excess of water and impurities, for checking the effects of too caustic lye, for shortening and hardening paste soap, and as filling material.

## CHAPTER IX.

### MACHINES AND UTENSILS FOR THE MANUFACTURE OF SOAP.

*Soap-boiling kettles or pans.* The older style of kettle, such as was usually employed in smaller plants for boiling soap over an open fire, consisted of two parts, of which the lower one, the actual kettle, of cast iron or copper, was set in masonry so that the heat acted upon the bottom. The upper portion, the so-called curb, was a hollow cone of iron-bound staves made to fit the flange of the lower portion, and could be made to extend as high as desired. The cone stood erect but had to be strongly and tightly fastened and joined to the lower portion. In this way a kettle could be enlarged at much less cost than a kettle wholly of iron.

The more modern kettles are of a circular, rectangular or square cross-section, but so far as practical working is concerned there is no material advantage of one form over the other. They are generally constructed of sheet-steel or wrought iron plates riveted together. Their dimensions vary according to requirement, one cubic foot being generally allowed for each fifteen pounds of fat to be saponified. Some kettles have flat, others convex or concave, bottoms. Fig. 11 shows a cylindrical kettle.

Direct fire heating is gradually going out of use in soap boiling it being employed only in small works. With an open fire the heating gases are generally allowed to pass under the bottom of the kettle and from there are conducted upward and around the kettle, and finally into the chimney flue. With the high temperature of the heating gases formed by the combustion of the coal, the bottom of the kettle suffers considerably if placed immediately over the grate, and scorch-

ing of the soap can only be prevented by very careful handling of the fire and constant stirring. This drawback is overcome by the manner of bricking in the kettles as shown in

FIG. 11.

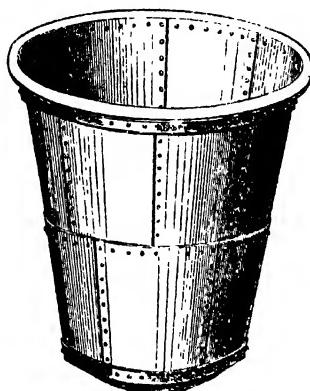


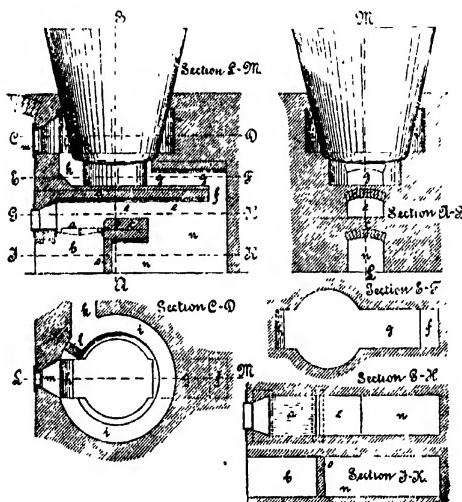
Fig. 12. The characteristic feature of this arrangement is a protecting arch placed between the bottom of the kettle and the grate, so that the flame, before it reaches the bottom of the kettle, yields up its strongest heat by passing under this arch.

The heating gases developed upon the grate *a*, below which is the ash-pit *b*, pass over the fire-bridge *c* into the channel *e*, which is covered by the fire-proof arch *d*; on the end of the channel *e*, the gases ascend upward into the vertical channel *f*, and finally reach the bottom of the kettle through the flue *g*. After passing under the bottom of the kettle they ascend upward through the inclined channel *h* into the lateral flue *i* running entirely around the kettle, and at *k* pass into the chimney flue. The circulation of the gases around the kettle is effected by the brick partition *l* built in the flue *i*. *m* is a hermetically closed manhole for cleaning the flues. The space *n*, located nearly behind the fire-bridge *c*, serves for the reception of flying ashes. It is closed at *o* by brickwork.

The above arrangement, which has proved very satisfactory in practice, permits the adjustment of any desired special contrivances, as, for instance, the smokeless combustion of the coal, discharge-cocks for the spent lye and soap, etc.

In large works steam is now, as a rule, used for heating soap kettles. In modern factories the soap is directly heated by means of steam circulating in a wrought-iron coil placed about

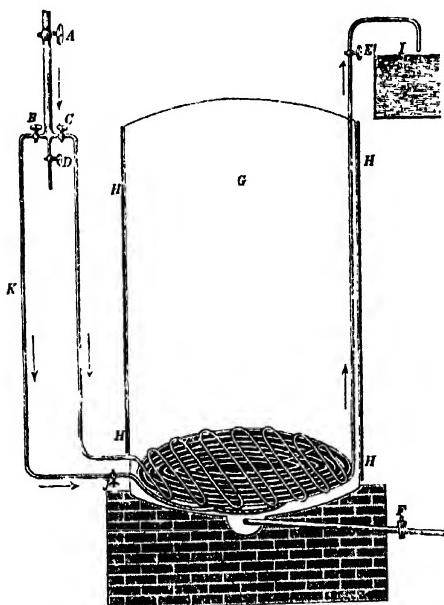
FIG. 12.



3 to 4 inches above the bottom. Fig. 13 shows such an arrangement with direct and indirect steam. *G* is the kettle of wrought-iron plates; *H H H* a wooden jacket about  $1\frac{3}{4}$  inches thick, to prevent too rapid cooling off in winter. *A* is a stop-cock on the main steam-pipe; *B* and *C* stop-cocks on the pipe system connected with the direct and indirect coils. *D* is a safety-cock; *E* cock for regulating the water condensing in the steam-coil; *I* lye reservoir; *F* discharge-pipe for the spent lye. The stop-cock *J* prevents the lye or soap from rising back into the steam-pipe *K* after the operation is finished.

The size of the pipes depends on that of the kettle; generally they have, however, a clear diameter of  $1\frac{1}{2}$  inches. The cocks *A* and *D* are provided for the sake of safety, so that in case the cocks *B* and *C* are not absolutely tight, the steam can nevertheless be prevented from entering the kettle. After finishing each boiling the cocks *A*, *B* and *C* are closed and the cock *D*

FIG. 13.



is opened. In case the cock *A* is not tight, the steam escapes from *D* without fear of danger.

Kettles of colossal size, capable of turning out 50 tons of finished soap at one operation, are by no means uncommon, and some of the large factories in this country have built even still larger kettles, requiring a building of three stories to contain them. Kettles whose contents are boiled by steam may be of

any desired shape, circular, oval or rectangular, provided that the steam-pipes be carried into the corners (if any) and be so arranged as to insure uniform boiling throughout the whole mass. There is no fixed proportion between diameter and depth; \* in England the diameter is to depth as 1 to 1, or 1 to 1.25, or 1 to 1.5, while in this country the ratio is increased as far as 1 to 2, 1 to 2.5, and 1 to 3.

To prevent loss of heat from the sides of the kettle, these may be covered with a non-conducting material, such as asbestos, or jacketed with wood, an air-space being left between the wood and kettle.

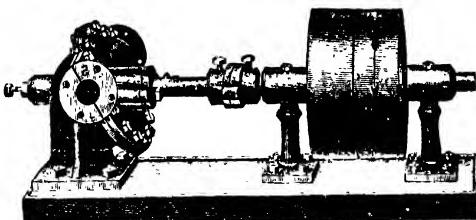
*Soap Pumps.*—The most convenient way of transferring the soap from one place to another, for instance, from the kettle or crutcher to the frames, is by gravity, but this plan is not always feasible, as it requires the various machines and appliances to be placed one above the other, and this can seldom be done. Some mechanical means have therefore to be employed for this purpose where the machines are on a level. In small works this may be done by hand labor with the use of dippers or ladles, but this plan is tedious and costly. It is, therefore, best to use a pump, especially made for the purpose.

A rotary pump, such as shown in Fig. 14 is most suitable for soap boilers. It may be set up in any convenient position adjacent to the kettle, and not over 10 feet above the bottom of the same, and connected to it by means of a  $2\frac{1}{2}$ -inch iron pipe, tapped through the iron plate at a distance of about 2 feet above the worm or coil. Each kettle is thus connected with the pump by the iron pipes, which have valves placed upon them on the outside (of the kettle), so that the contents of any one of the kettles may be readily pumped off and framed without disturbing the others. The pipe inside of the kettle has a suitable swing-joint so arranged that it can be raised and lowered at pleasure. Fig. 14 represents the pump

\* A kettle 15 feet in diameter and 15 feet deep will turn out 25 to 30 tons of soap.

complete. When the pump is rotated in the direction of the arrow, the outlet marked *S* is the suction; when rotated in the opposite direction, the opposite outlet becomes the suction. This is an important feature, as it enables the discharge-pipes

FIG. 14.



to be emptied of their contents in stopping, by giving a few revolutions by hand in the opposite direction. Fig. 15 is a view of the interior of the pump when the cover is taken off. When turned in the direction of the arrow the blade *F* sweeps around, drawing the fluid in at *I* and forcing it out at *H*, the contents of the pump being twice emptied during each revolution. The blade *F* swings on a pivot; the end of *F*, when reaching the point *B*, at the lowest point, gaining the position

FIG. 15.

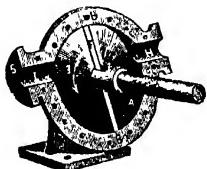
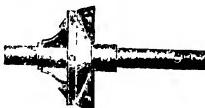


FIG. 16.



there shown, and gradually returning to its former position on completing the revolution. The fluid is prevented from passing from one side to the other by the contact of the cone with the cover. The set-screw shown in the illustration bears against a step at the end of the cone and keeps the cone forced

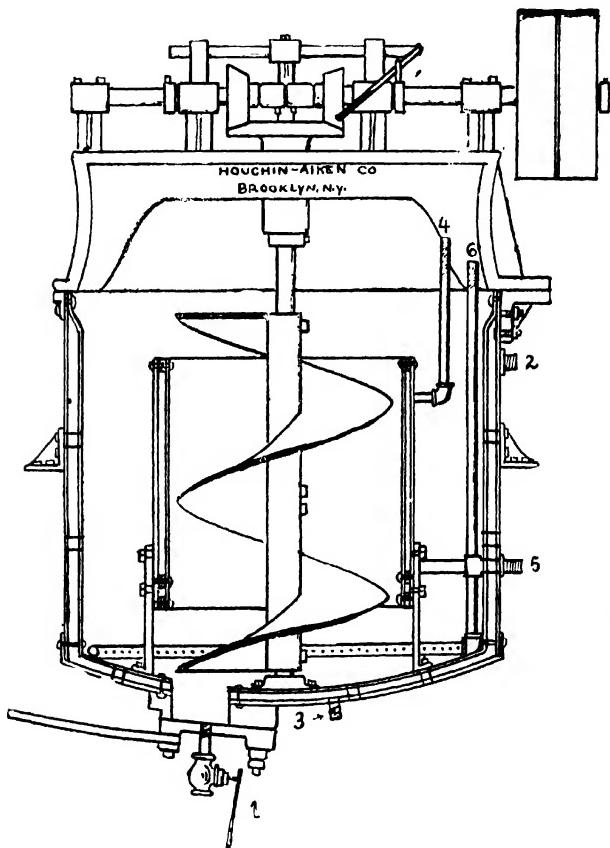
against the cover, and is screwed up to compensate for any wear that takes place. Fig. 16 shows the cone and blade and forms the entire working part of the pump. No valve being used, there is no chance of any derangement in the operation of the pump. The soap can be forced any distance or height. It can be pumped from one kettle into another, which is a special advantage when it is necessary to transfer the "nigre," either in the state of a soft curd or in the unseparated state, into another kettle to make room for a fresh boiling with clean stock, and thereby keep up a uniform quality of first-class soap. By lowering the pipe attached to the swing-joint inside of the kettle, the lye of the "strengthening change" can be pumped from the very lowest point in the bottom of the kettle, while still hot, into another kettle in which stock is being saponified, thereby economizing steam. In certain cases where it is undesirable to pump the lye over the curb into the kettle, because of the froth which it may occasion, another plan can be adopted, which admirably brings into play the whole system of pump, valves, and swing-joints of the two kettles, from the bottom of one of which it is required to pump the hot lye and force it through the iron piping down to the bottom of the other. Lye of any kind, whether spent lye or strengthening change lye, strong caustic lye, either hot or cold, grease or thick oil, can be easily and quickly pumped by this pump. The pump is 10 inches in diameter and  $2\frac{1}{2}$  inches in outlet, revolutions 120 per minute, capacity 6000 gallons per hour.

*Crutchers.*—In the early days of soap-making crutching was usually performed by hand with a utensil known as the crutch. The work was laborious and time-consuming and, while the hand-crutch is still used in some small works, it has been generally superseded by power-driven crutchers. The commonest form of such a crutcher is a circular vessel of sheet steel or boiler-plate. In the center of this is fixed a mixing apparatus, which usually consists of a central shaft which is made to revolve by suitable gearing. This shaft carries arms, the motion

of which through the mass of soap causes it to become mixed and form a homogeneous mass.

Fig. 17 shows in cross-section the "Perfection" crutcher

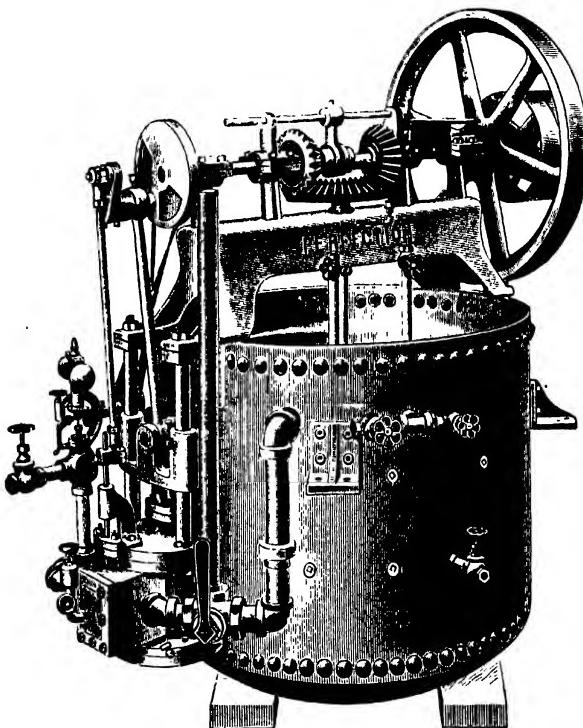
FIG. 17.



manufactured by Houchin-Aiken Co., Brooklyn, N. Y., and Fig. 18 the same crutcher fitted with a 10-horse power engine

for direct drive. The machine is made either with a plain shell and inner cylinder, or with a jacketed shell and inner cylinder. An iron valve  $5 \times 7$  inches, with scraped joint in the bottom, permits of the ready emptying of the machine.

Fig. 18.

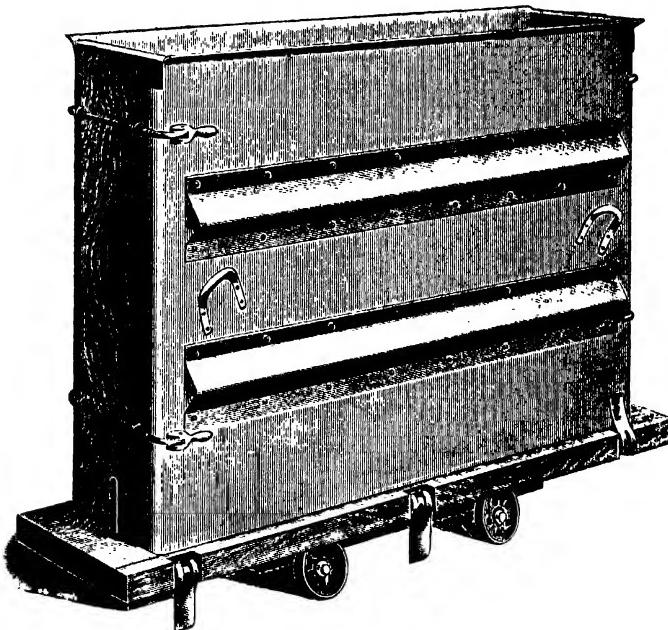


The jacketed machine, Fig. 18, consists of an outer shell made of two sheets of boiler steel, riveted together and stay-bolted so as to form a steam jacket, which extends under the bottom and up the sides of the machine; and an inner jacketed drum of similar construction, except that it is open top and bottom, is supported from the bottom and is slightly larger

than the mixing screw, which rotates in this inner drum, thoroughly mixing and crutching the soap. The mixing screw is driven through cut bevel gears, two pinions being provided with a clutch, so that the motion of the screw may be reversed. The jacketed shell and cylinder are tested for 100 lbs. working pressure.

*Soap frames.* This name is applied to the receptacles into

FIG. 19.

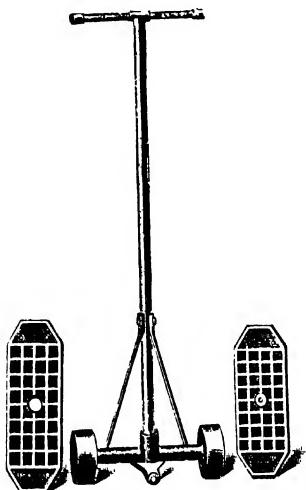


which the soap is run when drawn from the kettle, in order that it may cool. They must be so arranged that they can be easily and conveniently taken apart and put together and perfectly tight so as not to permit any leakage of the warm liquid soap. They ordinarily have the form of a parallelogram; their dimensions vary according to the quantity of soap

to be run into them, and are generally regulated by the capacity of the kettle. These frames were formerly constructed of wood and built up in sections, pine planks about two or three inches thick being as a rule used. The sections fitted closely upon each when piled in a series, and were tightly clamped from top to bottom. When the soap was cold the sections were lifted off, one at a time, and the soap remained upon the bottom ready to be divided into slabs.

The modern soap frames are generally made with sheet-steel sides and wooden ends and bottoms. Fig. 19 shows the

Fig. 20.



"Paramount" soap frame manufactured by Houchin-Aiken Co., Brooklyn, N. Y.

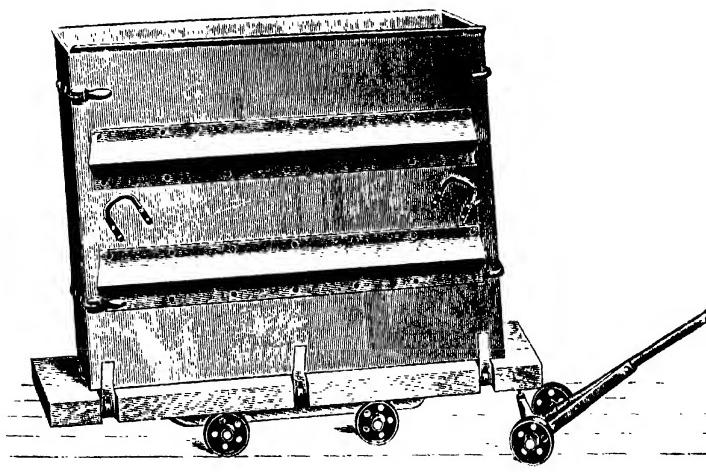
The ends of the frame are made of 2" x 4" pine, framed together with mortise and tenon joints and lined with No. 18 gauge steel, the steel being bent around the edges of the frame to form a metal seat for the metal sides. This arrangement gives a practically indestructible end, and which at the same time is light and which cannot warp or split, as is the case

with a solid end. If desired, solid ends are furnished, as shown on the illustration of the frame.

The bottoms are made of 3" seasoned hard wood, with 3" x 3" maple side rails and three eccentric bottom clamps with bolts, also two mild steel adjustable eyes for holding bottom to ends.

The bottoms are equipped with heavy cast-iron wheels, 5 $\frac{1}{2}$ " diameter by 2 $\frac{1}{2}$ " face, with 1 $\frac{1}{8}$ " square steel axles turned to

FIG. 21.



1 $\frac{1}{8}$ " for wheels. Each axle is bolted to bottom with two 2 $\frac{1}{2}$ " steel bolts with special heads. The axle rails are of 3" x 4" x 3' 6" spruce. Bottom plates are of 1" x 6" pine. Pivot plates furnished if desired.

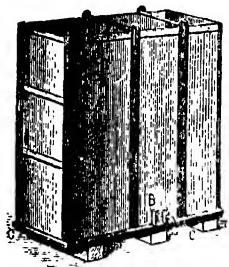
The sides are made of No. 12 gauge steel, with two No. 17 gauge steel reinforcing V's 3" high by 6" wide, and two mild steel handles on each side.

Fig. 20 shows the pivot truck and plates for soap frames. It is a patented device and enables the easy handling of filled

frames of soap by one man, and the packing of the frames closely together. Fig. 21 shows the application of the truck and plates to the soap frame.

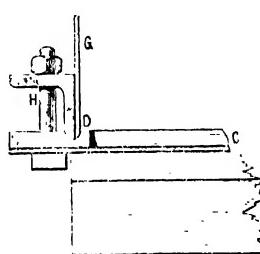
Soap frames are also made entirely of iron. They are best constructed of stout sheet-iron or sheet-steel properly strengthened to prevent bulging out. Figs. 22 and 23 show a frame made of sheet-iron about 0.17 inch thick and strengthened by flat-irons, angle irons and **T** irons. It consists of four side pieces and a bottom and can be taken apart. Its construction is as follows: The sides and bottom are sufficiently strength-

FIG. 22.



Iron Soap Frame.

FIG. 23.



Connection of the Sides with the Bottom of the Frame.

*A*, iron frame; *B*, brass-cock; *C*, bottom plate; *D*, grooves in the flat-irons; *G*, side; *H*, screws.

ened by angle-irons and stays of **T** iron. The actual iron bottom is fastened to a second wooden bottom of boards about one inch thick, the whole resting upon strong wooden beams, or upon wheels. The manner of stiffening the frame, especially the number and thickness of the stays of **T** iron, depends on the dimensions, frames with a capacity of 4400 pounds being, for instance, provided with four stays, those holding 8800 pounds with six, and so on.

The long screw-bolts on the top of the frame serve to keep the sides at a proper distance from each other, so that the

block of soap formed in the frame presents an exactly rectangular shape and perfectly level surfaces.

An absolutely tight and close joint is effected by the half-round and exactly planed edges of the angular irons fitting into grooves also exactly planed in the flat-irons as shown in Fig. 23. The sides are connected with each other and with the bottom by screw-bolts about  $2\frac{1}{2}$  inches long.

The frame is put together in the following simple manner: The workmen place first one of the long sides of it in the groove of one of the long sides of the bottom plate. While one of the workmen supports the side, the other fastens it to the bottom by pushing from below one of the short screw-bolts through each of the two bolt-holes and tightening the screw. One of the workmen then places one of the short sides, which he can easily carry, exactly in the groove of one of the short sides of the bottom, securing it to the bottom in the same manner as above and to the long side already in position, by pushing a screw-bolt through the upper and lower holes. The other short side is then secured in the same manner, and finally the second long side.

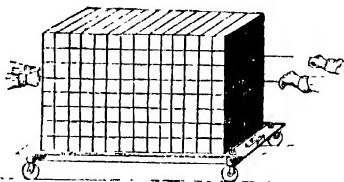
The frames for toilet soaps differ from those for ordinary soaps only in size.

For storing soft soap large wooden casks were, as a rule, formerly used, but iron holders have lately been introduced for this purpose, they possessing the advantage of saving space and being more durable. In a reservoir 3 feet deep, 3 feet long and 3 feet wide about 2000 lbs. of soft soap can be stored, and the weight of such a holder of wrought iron with sides 0.19 inch and bottom 0.23 inch thick, would be from 525 to 550 lbs. In the construction of such holders care must be taken to have the rivets smoothly countersunk in the interior so that in case lye has to be added to the soft soap, it cannot effect a lodgment. In iron holders the color of the soft soap is not impaired, while pale soaps frequently take up coloring matter from wooden vessels.

*Slabbing of soap.* After the soap has become sufficiently

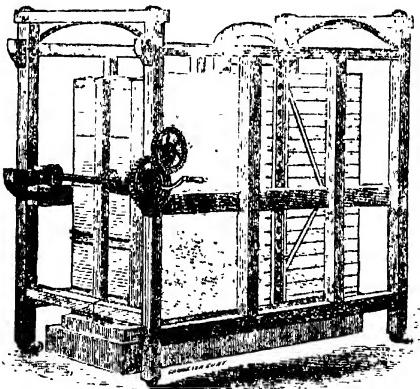
firm, the frames, according to their construction, are either lifted off or taken apart, so as to leave the solid block of soap resting upon the bottom. The block is then divisioned off by means of a scribe, an instrument of hard wood smoothly

FIG. 24.



planed and provided with slender iron teeth set at the thickness desired for the slab of soap. The workman then taking brass or steel wire fastened to a wooden handle draws it through the block, Fig. 24, which is thus divided into slabs.

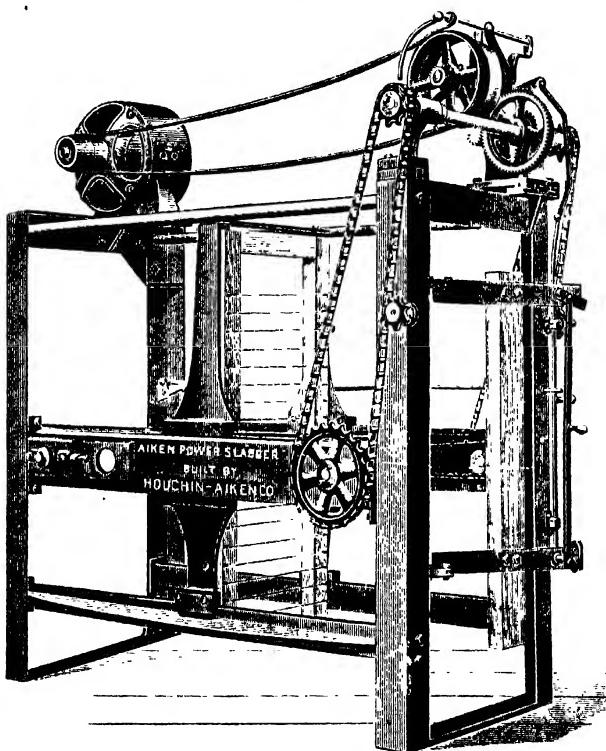
FIG. 25



When the whole is thus divided, the slabs are removed and reduced in their turn to bars and lumps of smaller dimensions. The workman is provided with a long piece of steel wire, fastened to two handles, or in some instances to one.

This wire is fitted to the mark made by the scribe and then drawn through, a slab of soap being thus divided from the main block. The operation is repeated until the whole block has been cut into slabs. This mode of slabbing is quite sim-

FIG. 26.



ple and is still employed in some factories, but in most works it has been superseded by the use of the slabber.

Fig. 25 shows a hand-slabbing machine. The method of operation will be readily understood from the illustration.

The machine has a movable head and the wires are drawn through the soap by compound gearing with a steady pull, all the while in operation, thus leaving the slabs perfectly level and smooth, each and every one of the same thickness, with a fine luster, after the wires have passed through the soap.

The illustration shows the soap in the machine and the wires partly drawn through. When cut through, the hinge on the back end is opened, the machine drawn from the soap, then the head is run back, the shaft placed in the boxes above, the hinge folded in front and drawn to another frame, then the hinges are closed, the shaft is placed in the former boxes, and the head-block placed between the shaft and the front of the soap, when the machine is ready to cut another frame. The whole process for getting ready and cutting a frame of soap can be done in a few minutes.

Fig. 26 shows the "Aiken" power slabber, manufactured by Houchin-Aiken, Brooklyn, N. Y. This machine is constructed wholly of iron and steel and is fitted with interchangeable cutting heads, which can be removed by loosening four bolts. It is built for direct motor drive, as shown in the illustration, or for belt drive. The machine is self-contained, and when bolted to the floor is ready for the belt or motor. This machine automatically cuts the soap, then ejects the cut frame, the cutting head returning to its initial position and stopping, ready for the next frame. The machine will cut anything that the wires will hold.

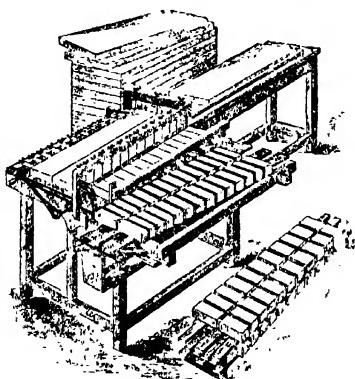
*Cutting tables.* After the soap has been cut into slabs, the latter are cut into bars, a number of machines, to be worked either by hand or power, having been devised for this purpose.

Fig. 27 represents a machine for cutting, trimming, racking and spreading the soap ready for drying, leaving all the bars or pieces of an equal size and weight, smooth and clean, without any finger marks.

First the slab is placed on the table, then drawn through the wires endwise, trimmed, and then passed through wires that cross-cut it into bars, and then left on the racks, which,

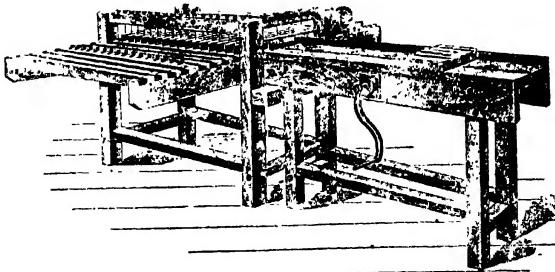
by being drawn endwise, spreads the soap in a uniform manner, all the bars at equal distances apart, ready for setting away to dry and box.

FIG. 27.



A "double rack" hand cutting table, manufactured by Houchin-Aiken Co., Brooklyn, N. Y., is shown in Fig. 28. The operating mechanism is of iron and steel and so designed

FIG. 28.

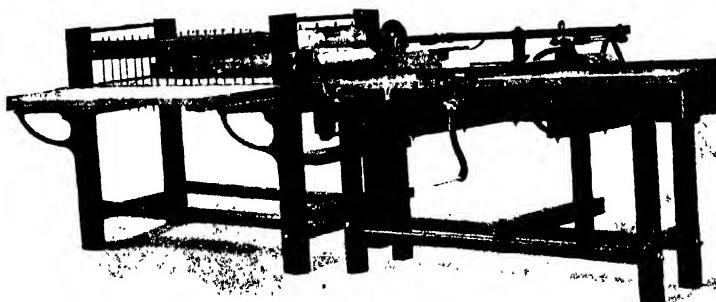


that a single crank operates either the wide or narrow pusher as desired. The pushers are operated through steel chains and racks, the cut cakes being automatically spread and sepa-

rated on the racks, the scrap being automatically removed. The cutting heads and pushers are interchangeable and can be very quickly removed and replaced with other heads and pushers. The machine is adapted to cut square, oval, round-cornered or octagon-shaped cakes. The rack holder is removable, so that if it is desired to stamp the cakes by hand, a table can be substituted in place of the rack holder, or the soap can be stamped on the rack holder if desired.

This machine works to best advantage when using the double rack, which is of double the width of the ordinary rack, and is designed for holding one slab cut into cakes and

FIG. 29.



separated. It is usual with this rack to have special dry-room trucks, built to take one tier of the double racks. Great economy of handling is found with this arrangement.

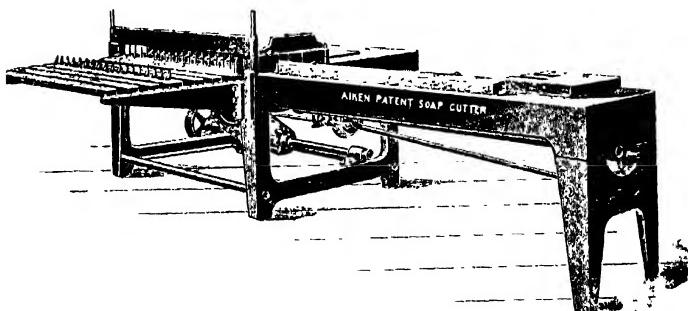
The combined cutter and stamper shown in Fig. 29 is similar in construction to the previously described machine, but in addition it is fitted with a die for stamping the cakes as the soap is cut. It can be operated either by hand or power, and used either with or without the stamping device as desired.

The "Aiken" power cutting table, Fig. 30, which is also manufactured by Houchin-Aiken Co., Brooklyn, N. Y., is a

patented device. It automatically cuts the soap into cakes, then spreads and separates the cakes on the racks. It is driven by power, is built entirely of iron and steel and is strong, but simple in design. It has interchangeable steel cutting heads and pushers. Any shape of cake desired can be cut and only from 10 to 14 seconds are required to cut each slab into cakes and spread and separate the cakes on the racks. The machine is particularly adapted for cutting oval-shaped cakes, special cutting heads being provided, which replace the ordinary heads.

The reciprocating motion on the pushers is obtained through

FIG. 30



a chain drive under the table, operating automatically, bringing the pushers to a stop on their forward movement with a uniformly retarded motion, and then increasing the speed on the return from zero to the maximum speed with a uniformly accelerated motion, thus giving an easier movement with greater speed than when the reverse motion is obtained abruptly. The machine may be operated economically with either two, three or four men, the output varying with the number of operatives employed.

*Soap presses.* While laundry soaps were formerly, and are yet, sold in bars without being stamped, they are now largely brought into commerce in the form of cakes, and these are, as

a rule, stamped. However, as it is almost the universal custom to stamp toilet soaps, the presses used for the purpose will be referred to in the chapter devoted to that branch of the industry.

*Drying of soaps.* While some soaps do not require drying, others do, and it is especially desirable that cakes should be dried a little before stamping so that a thin crust of comparatively hard soap is formed, as then a better impression is obtained. The drying room used for this purpose may be heated by steam or warm air, or be well ventilated by air. The latter does not require any heating apparatus but can only be used in warm weather. It is generally located in the upper story of the factory where the air circulates freely. Shelves or racks on which are placed the pieces of soap to be dried, are fixed in the room eight or ten inches apart, one above the other, this separation having the advantage of accelerating the drying of the soap by putting it in contact with a greater mass of air. The desiccation is more rapid when the temperature of the air is elevated. This mode of drying is the most economical, requiring neither apparatus nor fuel; it is also the most regular and the best for the drying of soap, and it may be used whenever circumstances will permit. Unfortunately it is, however, subject to the variations of seasons and weather so frequent in our climate. The drying-rooms with warm air have the advantage that they can be used at all seasons. In many factories the drying-room consists of a more or less large room around which shelves provided with trays are disposed and upon which are placed the pieces of soap to be dried. In the middle of the room is a stove heated with wood or coal. The temperature must not be above 80° F.; openings must be made in different parts of the room to permit the air saturated with moisture to escape freely. This arrangement quickly hastens the drying of the soap. A temperature of 80° F. is sufficient to dry in fifteen or twenty hours pieces of olein soap intended to be moulded.

But this mode of drying presents the inconvenience of local-

izing and causing an unequal distribution of the heat. Some shelves are remote from the source of heat, so that the soap does not dry equally in all parts of the room. And, besides, stoves often smoke, especially when first lighted, and the smoke stains and blackens the pieces of soap. These drawbacks are overcome by the use of a drying-room heated by hot air. By this system the heat produced by the fuel is completely utilized and the hot air which flows into the room is always pure, without either odor or smoke. The chief advantage of this system is that the desiccation of the soap is rather produced by an energetic ventilation occasioned by the abundance of hot air continually renewed in the room, than by a high temperature.

For drying toilet soaps, sheet-iron ovens furnished with a grate and best heated with coke may be used. They are made with double walls, between which the hot air passes, thus uniformly heating the interior chambers. The gases from the fire escape through a flue fixed at the top of the oven, which can be opened or closed to regulate the temperature.

## CHAPTER X.

### PRODUCTION OF SOAPS.

SOAPS may be divided into two principal groups, viz., hard or soda soaps, and soft or potash soaps. The first group comprises curd or grained soaps, half-grained soaps and paste soaps. The grained soaps show a characteristic crystalline formation termed "curd" or "grain." They are prepared either by freeing the alkaline oleate from superfluous water and glycerin by the aid of common salt (salting out), or by not completely salting out, by adding only sufficient common salt, or using an excess of lye, so that a precipitate of paste is formed from the soap at rest. This latter kind of grained soap can only be prepared by the use of cocoanut or palm-kernel oil in connection with other fats; it is technically known as *grained soap upon a precipitate of paste*, or *deposited grained soap*, while the first variety is termed *grained soap upon spent lye*. The grained soaps, when again heated on water or weak lye, are known as *ground grained soaps*.

*Paste soaps* are obtained by simply allowing the soap-paste to congeal. They are prepared with the aid of cocoanut-oil and palm-kernel oil. They contain all the glycerin present in the fat subjected to saponification and a large amount of water. They are either smooth or exhibit but little formation of grain, which, if mottled soaps are to be prepared, can, however, be brought out stronger by the addition of coloring substances such as Frankfort black, ultramarine, etc.

*Half-grained soaps* (in Germany known as *Eschwege soap*) can only be prepared with the aid of cocoanut and palm-kernel oils; they exhibit some formation of grain which may also be brought out stronger by the addition of coloring substances. These half-grained soaps are boiled either by the direct or the

indirect method. The first process consists in boiling cocoanut oil or palm-kernel oil together with other fats, such as palm-oil, bone-fat, fuller's fat, etc., and the second by preparing a grained soap from one or several of the last-named fats, and adding to this a paste soap boiled from cocoanut oil or palm-kernel oil.

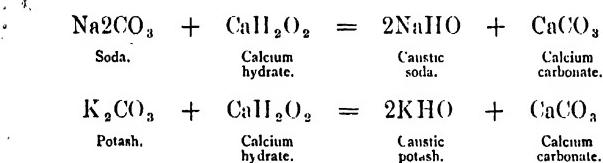
*Potash or soft soaps* are divided into three principal varieties : (1) *transparent soap*, variously colored and brought into commerce under different names, such as oil soap, crown soap, green soap, black soap, glycerin soft soap, etc.; (2) *natural grain or fig soap*, which is transparent and shows a "figged" appearance; (3) an opaque soap of a white or yellowish-white color, designated as smooth *olein soap*, *silver soap*, etc.

Before entering upon a description of the methods of boiling the various kinds of soap found in commerce, the preparation and examination of lyes will be discussed and a few general remarks be made on the process of boiling.

*Preparation of lyes.*—For the saponification of the fats, the correct constitution of the lyes is of the utmost importance, and, hence, their preparation is a serious problem for the soap boiler. With the use of caustic soda or caustic potash the operation is quite simple, they only requiring to be dissolved in water to the desired degree. On the other hand, the preparation of lyes from alkaline carbonates requires great care. Fats, except when they are rancid and consequently contain free fatty acids, are not decomposed, even at a boiling heat, by alkaline carbonates, and the latter, for the saponification of neutral fats, have first to be converted into caustic alkalies. To be sure, many soap boilers are of the opinion that the alkaline carbonates in the lye also exert a saponifying effect upon the fats, and endeavor to prove it by the fact that many fats can only be saponified with difficulty with pure caustic lyes, and that for many soaps a content of alkaline carbonate in the lye is required. This view, however, is not correct. The alkaline carbonate, sure enough, frequently plays a role in saponification, as well as in the soap, but only

a mechanical and not a chemical one, which is shown by the fact that in all cases where it appears necessary, it can be replaced by sodium chloride, or potassium chloride, or other salts. A neutral fat can only be saponified by caustic alkali, and every neutral fat can be saponified with it as soon as the lye has the concentration suitable for it, and saponification is effected at an appropriate temperature.

*Slaked lime—calcium hydrate*—serves, as previously mentioned, for causticizing the alkaline carbonates. The carbonic acid combines thereby with the lime, while the alkali takes up the hydroxide:



This chemical process takes place, however, only when dilute solutions of the alkaline carbonates are treated. The calcium carbonate is insoluble in water and settles in the form of mud to the bottom, while the caustic alkali remains dissolved in the lye. This process does not take place with more concentrated solutions of soda or potash. Liebig, on the contrary, has pointed out that a more concentrated caustic potash solution decomposes the calcium carbonate, and consequently calcium hydrate, in the presence of little water, cannot decompose the alkaline carbonate. For the preparation of perfect caustic lyes, the potassium carbonate has to be dissolved in at least 10 to 12 parts of water, and the sodium carbonate in 8 to 10 parts.

The preparation of lyes is best and most quickly effected with the assistance of heat. Lyes, to be sure, may be causticized in the cold way, but more lime and more dilute solutions of alkali have to be used, and both have to act upon each other for a longer time.

In plants where steam is available the preparation of lye is generally effected as follows: Waste lye from a previous operation is brought into the converting kettle and heated by the admission of steam to 167° F. The alkaline carbonate to be causticized is then introduced and its solution effected by crutching. The lime required is then gradually added in small portions to the hot solution, best by means of the lime basket—an iron box with a perforated bottom—suspended over the kettle so that it dips partly into the solution. The lime slakes in the basket and reaches the kettle in a finely divided state, so that an intimate mixture with the solution of alkaline carbonate is effected. The first lye, in the case of soda, is generally taken at a density of 24 to 25° B., the second at from 18 to 20° B., and the third at from 12 to 15° B.; and in the case of potash, the first at from 28 to 30° B., the second at 20° B., and the third at from 14 to 15° B. The different grades of soda lye are kept separate in special holders so that a supply of lyes of varying densities, as may be required, is always on hand. The different grades of potash lye are also sometimes kept separate, but this is frequently not done, and an average lye of a density of about 20° B., such as is suitable for boiling soft soaps, is made by mixing the different grades. The lime-mud which remains in the kettle after taking the third lye, is washed with water until a sample of the liquor shows 0° B., a proof that all the alkali has been withdrawn from the lime. This waste lye is utilized in the next operation for dissolving the soda or potash.

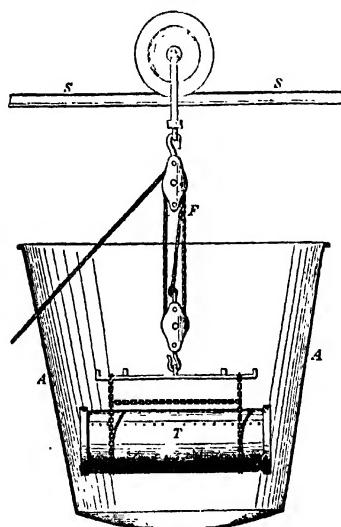
Where steam is not available it is advisable to heat the waste lye to boiling in a special kettle and bring it hot into the converting kettle containing the alkaline carbonate; solution of the latter is promoted and quickly effected by crutching. The lime required is then added, and by its slaking the liquor is again brought to the boiling point. The remainder of the operation is the same as above described.

The preparation of lye from caustic soda or potash is a very simple operation. The contents of the drum are best dissolved

with the assistance of a device shown in Fig. 31. An iron rail, *S*, is secured in the walls of the factory so as to run about  $6\frac{1}{2}$  to  $7\frac{1}{2}$  feet above the edge, and directly over the center, of the converting kettle, *A*. Upon this rail runs a large cast-iron pulley (about 12 inches in diameter), to which is fastened the tackle *F*. The lower end of the tackle is provided with a strong iron hook.

The drum *T* to be emptied is first pounded on all sides with

Fig. 31.



a heavy iron hammer, which causes the soda to crack. Holes the size of a hand are then cut with a hatchet in the head and bottom of the drum near the edges. Two chains are next slung around the drum and suspended to a horizontal iron rod, as shown in the illustration. The drum is then hoisted to above the edge of the kettle by means of the tackle, and, after pushing forward the pulley upon the iron rail so that it is over the center of the kettle, the drum is lowered to about

one-half the height of the kettle, and the tackle secured. Water is then admitted to the kettle, and the drum from time to time raised to permit the dissolved soda to run out. The entire work, including the dissolving of the soda, is effected at the utmost in two hours, about ten minutes of that time being taken up by preparing and placing the drum in position. The solution is then made up to the desired degrees. The quantity of caustic soda or caustic potash which is dissolved at one operation should be large enough to insure a supply of lye for eight to fourteen days, this depending of course on the quantity of fat which is to be saponified during that time. An allowance of 20 to 21 lbs. of caustic soda for 100 lbs. of fat and 28 to 29 lbs. of caustic potash for 100 lbs. of oils and fats will be amply sufficient.

The residue which finally remains in the converting kettle, may be utilized in various ways. If derived from soda it is diluted with 3 to 4 parts of water; the resulting dirty turbid lye of 13 to 16° B. being used for boiling grained soaps which are subsequently salted-out. Or, the residue is brought into an iron reservoir and a bucketful of cold water poured over it. The whole is then allowed to stand without being crutched. A clear, though of course weaker, lye is obtained and may be utilized for every kind of soap. The residue derived from the preparation of caustic potash lye is also utilized in this manner.

Caustic potash lye previous to being used is diluted with water, or if it is to be employed for soft soaps, with potash solution. A suitable lye is obtained by diluting 100 lbs. of lye of 50° B. with 200 lbs. of water and 70 lbs. of potash solution of 30° B.

The strong lyes obtained by causticizing with lime when drawn from the converting kettle still contain a considerable quantity of alkaline carbonate; only the lyes of a density below 14° B. may be entirely caustic. Since soft soaps are not simple neutral soaps, but require for their consistency a certain quantity of salts, perfectly caustic lyes are not required for

boiling them. Of the high-grade soda lyes those with a density of 20 to 24° B. are suitable for boiling half-grained soap and certain paste-soaps, which also require salts for their formation, while those with a density of 16 to 18° B. are serviceable for the saponification of fats which contain much free fatty acid, such as palm oil, bone fat, etc., and for that of fatty acids such as olein, etc.

The soap boiler will at the present time find it seldom necessary to prepare perfectly caustic lyes by causticizing alkaline carbonates, as he can more conveniently obtain them by dissolving caustic soda or caustic potash. For certain kinds of soap a perfectly caustic lye is not only not required but a certain content of alkaline carbonate is even desirable, as otherwise it has to be added in some other way. Some soap boilers designate their lyes as *just right in lime, too high in lime, or too low in lime*. In the first case it means that in preparing the lye the correct quantity of lime has been used, that the quantity of caustic alkali in the lye is in right proportion to alkaline carbonate for boiling a certain kind of soap. In the second case, too little lime has been used, or the alkaline carbonate solution has been too concentrated and consequently the lye is not sufficiently caustic. The third case, on the other hand, means that the lye is too caustic for the preparation of certain soaps.

The expressions "too high in lime," "too low in lime," etc., are also frequently applied to the soaps themselves. However, this does not imply that the soap contains too much or too little lime, but that the lye used has been too caustic or not sufficiently so.

*Examination of lyes.*—In many soap factories the lyes are only tested by means of a hydrometer, which is a very convenient method but gives rise to considerable errors. The hydrometer simply indicates the specific gravity of the lye, and this is affected by all the substances dissolved in it, while the soap boiler is only interested in knowing the content of potash or soda. If he had only to deal with solutions of pure

soda or potash, testing with the hydrometer would suffice, but as the lyes contain chlorine combinations, sulphates, etc., two lyes showing the same degree by the hydrometer may contain varying quantities of caustic alkali or of alkaline carbonates. The only lyes with which this cannot happen are those with a density of over 35° B., because carbonates and other salts are no longer soluble in them. If soda containing 90-per cent. sodium carbonate be dissolved in enough water to form a solution of 15° B., it is evident that the latter contains more sodium carbonate than a solution of the same density prepared from soda containing 75 per cent. sodium carbonate. Now if only so much lime is taken as is required for the causticization of the quantity of sodium carbonate contained in the weaker solution, it does not suffice for the more concentrated solution, and more soda remains fixed on carbonic acid. For soda or potash of known quantity the hydrometer may possibly serve as a guide, but not when working with unknown materials. This is perhaps the reason why many soap boilers are reluctant to make a change in their brands of potash or soda. They frequently work poorer and more expensive material because they are used to it.

To be successful with soaps, it is of the utmost importance that the quantities of caustic alkali and alkaline carbonates are in correct proportion contained in the lye. The soap boiler should therefore not depend alone on the Baumé hydrometer, but test the lyes as to their content of alkaline carbonates and caustic alkali.

By simply titrating the lyes, the entire content of alkaline carbonate and caustic alkali is determined either according to the calculation as alkaline carbonate or caustic alkali; however, the content of each of them can be readily established in the alkalimetric way by first determining in one sample the total content, and then separating in another sample the alkaline carbonate with the assistance of barium chloride solution,\*

\* Barium chloride combines with the alkaline carbonates to barium carbonate, which is insoluble in water, and to soluble alkaline chlorides upon which the test acid does not act.

and determining the quantity of caustic soda in the liquor filtered from the precipitate. The difference between the first and second determinations gives the quantity of alkali which has not been causticized.\*

If, for instance, a high-grade causticized soda lye is to be tested and a very accurate scale is not available, weigh out 40 grammes of the lye in a beaker, then bring the lye into a liter-flask, rinse the beaker with water, pour the rinsing water into the flask, and fill the latter up to the mark. One liter of liquor then contains 40 grammes of lye, hence 100 cubic centimeters = 4 grammes. Now carefully bring by means of a pipette 100 cubic centimeters from the flask into a beaker, color blue with litmus tincture, and then allow to run in from a burette enough standard acid to color the liquor permanently red after boiling. Now, suppose 21.5 cubic centimeters of standard acid have been used for this purpose. For the determination of the quantity of acid added in excess carefully add from a burette standard alkali solution until the liquor appears perceptibly blue. Suppose the quantity of standard alkali solution used in this manner amounts to 1.5 cubic centimeters, then  $21.5 - 1.5 = 20$  cubic centimeters of standard acid have been required. Since 1 cubic centimeter of this acid is equivalent to 1 per cent. caustic soda, the lye contains 20 per cent. caustic soda, provided it contains the alkali only as caustic soda.

Now bring once more from the liter-flask by means of a pipette 100 cubic centimeters into a beaker and add barium chloride solution until no more precipitate is formed. After allowing the precipitate to settle, filter through a paper filter in a glass funnel, and wash the contents of the filter with distilled water till the liquor running from the funnel no longer colors red litmus paper blue. Then color the filtrate blue with litmus tincture and titrate as before; boiling, however, is not necessary as no carbonic acid which might impair the

\* Compare p. 161, "Testing of Soda and Potash."

appearance of the color is present. Suppose in the second titration 16 cubic centimeters of standard acid have been used, then the lye contains 16 per cent. caustic soda. The difference of 4 per cent. between the first and second titrations is the quantity of soda which has escaped causticization. In the first titration this has been accounted for as caustic soda, though it was present as carbonate, and it must therefore be reduced according to the following equation :

$$40 : 53 = 4 : x = 5.3.$$

Hence the lye contains 16 per cent. caustic soda and 5.3 per cent. sodium carbonate.

For the examination of potash lye the process is the same, except that 50 grammes of it are weighed out and the liter flask is filled up to the mark. The total content of potash is then determined in 100 cubic centimeters, and in another 100 cubic centimeters the caustic potash after the potassium carbonate has been separated by barium chloride solution. From the difference between the first and second titrations the potassium carbonate is calculated, taking into consideration that caustic potash to potassium carbonate is in a ratio of 56 to 69.

To test whether a lye is perfectly caustic, pour a clear sample of it into dilute acid; no effervescence should take place. The lye should be poured into the acid, not the reverse, as in the latter case incomplete neutralization may readily occur and no carbonic acid is evolved notwithstanding the presence of potassium carbonate in the lye.

#### BOILING OF SOAPS.

The saponification of fats by the alkalies is in most cases effected by boiling, saponification below boiling heat being, as a rule, applied only to cocoanut oil. However, it has been shown by Mège-Mouriès that under certain conditions the saponification of other fats can also be effected below boiling

heat. If melted tallow is emulsified by shaking with white of egg, gall, or a similar substance, and in this state mixed with potash or soda lye, and allowed to stand for three or four hours at a temperature of from 113° to 140° F., it is, at a temperature of above 140° F., completely converted into soap. According to Knapp, all fats possess this property even at an ordinary temperature, and without the use of white of egg or a similar substance. By pouring a few drops of caustic soda into a bottle, then allowing it to run out and rinsing with water, every variety of fat brought afterwards into the bottle and shaken with half or equal its volume of water, is readily converted into an emulsion, for instance, oil with one-half or an equal volume of water. Solid fats, such as tallow, require a certain degree of heat to render them fluid, and must only be shaken as long as the temperature does not sink below this point, as otherwise they separate in lumps like butter in beating cream. Such emulsions can be diluted at pleasure, and allowed to stand for days without separating into a coherent layer of fat and water, a thick cream only collecting over the thinner milky liquid. Under the microscope such an emulsion appears as a multitude of transparent globules of fat of a very small diameter floating in the clear fluid. The emulsion is, therefore, solely a fine division of the fats similar to the grinding or pulverizing of other substances. By mixing the undiluted emulsion with an excess of caustic lye and allowing the mixture to stand a few hours, the milky condition disappears and the whole separates into clear lye upon which floats a coherent cake of soap. This cake of soap is, according to the strength of the lye used, either hard and brittle, or soft and more jelly-like. As regards the rest, the strength of the lye exerts no influence whatever, provided it is not below three per cent. Hence, with finely divided fats saponification takes place with great ease in the cold. Melted or naturally fluid fats mix in their ordinary condition with lye to an emulsion-like mass, but saponification takes place with greater difficulty. 100 parts by weight of lard mixed with 400 parts

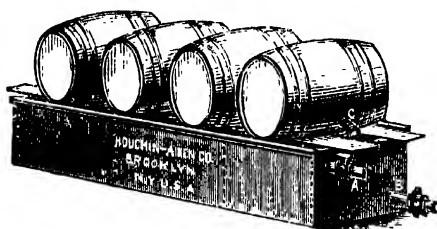
by weight of water and 60 parts by weight of caustic potash were only converted into soap after digesting for two days at a temperature of from 158° to 194° F. (Chevreul).

Furthermore the process of saponification is not even accelerated, as might be supposed by boiling with lye. The fat, to be sure, soon combines with a portion of the alkali, but, as a rule, a complete soap is only formed by gradual absorption, in the way of a continuous saturation, sometimes only after boiling for several days. The strength of the lye is of the utmost importance for the success of the operation; most fats can only be readily saponified by boiling if the operation is commenced with weak lyes, and the strength of the latter is gradually increased. The reason for this difference in behavior of emulsified and non-emulsified fats is evidently found in the fact that with the use of an emulsion the globules of fat float isolated in the lye, the fat thus presenting a number of attacking points to the lye. With the use of a boiling heat the fat melts and forms a coherent mass which spreads out over the lye; hence there is not the same intimate mixture of fat and lye, and saponification is not so readily effected. The reason why weaker lyes induce saponification with greater ease than stronger ones, may be explained by the fact that soaps are insoluble in strong lyes. To be sure, quite strong lyes are now frequently used for the preliminary boiling, but this is only possible when they still contain much alkaline carbonate. Cocoanut oil and palmkernel oil are, however, exceptions in this respect, they requiring strong caustic lyes for saponification.

The reason why almost all soaps are prepared by boiling, notwithstanding that saponification is not promoted by it, is that the mass formed by the combination of fat and lye requires at least an evaporation of the excess of water to make it into soap, an operation best effected by boiling. Every soap-boiler knows that strong boiling does not promote saponification, and for that reason insists upon a moderate fire in the preparatory boiling.

*Boiling with steam.*—The operation of boiling is mostly carried on in open kettles heated by fire or by steam, the latter being the most modern plan. In plants where steam is available, it is not only used for boiling soap, but also for emptying barrels by melting the fats contained in them by its aid, and for the preparation of lyes. Fig. 32 shows the principle of a steamer for emptying barrels, hogsheads, etc. By its use the waste by the old method of digging out the tallow, cocoanut oil, etc., is avoided, and the package is not destroyed or damaged in the least and is left perfectly clean. The stock is also cleaned, all the dirt settling to the bottom of the receiving tank, along with the water from the steam. The stock is also hot when run into the soap kettle, saving time and steam.

FIG. 32.



Either direct or indirect steam is used for boiling soap. In boiling with direct steam stronger lyes are required than with indirect steam or with an open fire, so that not too much water gets into the soap. Superheated steam may in this case be recommended, but is not absolutely required. Boiling with direct steam in large kettles answers very well, but, with indirect steam under the same conditions, proper boiling does not take place, as in large double or jacketed kettles the soap boils only near the walls of the kettle, and with steam coils only close by the steam pipes. Indirect steam has proved a success only with small, double boilers for the production of toilet soap, half-grained soaps and paste soaps.

The method of boiling with direct steam and an open fire, which is frequently employed, has proved very satisfactory.

With the use of direct steam the latter enters through a pipe-system on the inner wall of the kettle. By steam the stock of fat and lye is more rapidly brought to the boil than is possible with an open fire under the kettle. As the combination between fat and alkali takes place very rapidly, the contents of the kettle may sometimes show a tendency to boil over, but this can soon be curbed by shutting off the steam. The steam which passes into the kettle of course carries along considerable moisture, and this has to be taken into consideration as regards the strength of the lye to be used, *i. e.*, lyes of much greater density have to be employed than in boiling over an open fire.

The soap paste which is very soon obtained by the action of the steam has to be either salted out or evaporated. For salting-out it is best to use spent lye from a former operation in which additional salt is dissolved. With the use of dry salt the operation requires more time, the reason for this being that the salt immediately falls to the bottom of the kettle. In boiling with an open fire, the salt, by the required stirring is again raised and soon dissolved, but in boiling with steam there is no stirring and consequently it takes much longer for the salt to dissolve.

For preparatory boiling and boiling to grain soaps which are to be salted out, the use of steam is the quickest and surest method. It has the further advantage of the soaps thus produced retaining their light color, as they cannot suffer damage from the heat of the fire. After drawing off the spent lye, an open fire is, however, again used in order to prevent the incorporation of unnecessary water which would later on have to be evaporated. The application of direct steam is in order only in the case of fitting ground grained soaps and, when lye of sufficient strength has been used, such soaps can frequently be entirely finished, especially when lye of caustic soda is employed, the grain, when salted-out, being then often obtained

perfectly free from froth. The water formed by the introduction of the steam effects here a gradual grinding of the soap.

For boiling with indirect steam the kettle is fitted with a steam coil running in several turns along the walls of the kettle and finally terminating outside. Thus no steam or water formed by condensation enters the kettle. The end of the steam-coil is provided with a stop-cock for regulating the passage of the steam through the coil. The lowest turn of the coil is about  $5\frac{1}{2}$  inches above the bottom of the kettle.

For boiling with indirect steam a higher pressure is required than with direct steam. The mixture of fat and lye comes readily and quickly to the boil and, as in boiling with direct steam, a tendency of the contents of the kettle to rise can be checked by shutting off the steam. The formation of water by the condensation of steam being here avoided, lyes of the usual strength can be employed. For soaps which are to be salted-out, this method of boiling is all that can be desired, the operation proceeding smoothly and regularly, as the lye can always be kept at a uniform boil.

The case is, however, different when boiling half-grained, smooth white-grained, or soft soaps. These are soaps from which the moisture required for combination has to be again partly evaporated. So long as the soaps are thin, *i. e.*, contain a large amount of water, everything proceeds smoothly, but as soon as the soap thickens, it is impossible to further evaporate them with the assistance of indirect steam. Caustic potash lye of  $50^{\circ}$  B. being at present available, soft soaps are also frequently boiled with indirect steam. The caustic potash lye is, according to the season of the year, mixed with 16 to 20 per cent. of 98 per cent. calcined potash, and the lye made up to  $33^{\circ}$  B. Saponification proceeds well and the process has proved a success as, owing to the high-grade lyes, no water has to be evaporated.

In this country where the kettles are provided with direct as well as indirect steam, the procedure of boiling rosined grained soaps is as follows: When the fat together with the

rosin lye has been brought into the kettle, direct steam is introduced or the mixture is brought to boiling by indirect steam. When the mass boils, the operation is continued with direct steam, whereby the contents of the kettle are kept in constant undulating motion, and lye is then added until the paste becomes clear. Salting-out is then proceeded with in the usual manner and, when the salt is dissolved, the direct steam is shut off and boiling with indirect steam continued till a sample of the lye is perfectly clear and thinly-fluid. After the time required for settling, the spent lye is drawn off through a stop-cock on the lower part of the kettle. The lye required for clear boiling is then brought into the kettle and boiling with indirect steam continued till the soap possesses the characteristics of a well-boiled grained soap.

Closed apparatus is sometimes used for boiling soap in this country, as well as in England. It has the drawback that the course of the process cannot be sufficiently watched.

#### SEVERAL MORE MODERN PROCESSES IN THE MANUFACTURE OF SOAP.

*Saponification by alkaline sulphides.*—Pelouze has observed that the alkaline sulphides, used in the same manner as caustic alkalies, possess the property of saponifying fats. At an ordinary temperature saponification takes place in five to ten days, but immediately with the aid of heat. In the latter case hydrogen escapes and one equivalent of sodium sulphide yields the same quantity of soap as one equivalent of anhydrous soda.

In regard to this method of saponification, Dullo remarks that though saponification is quickly and completely effected, he cannot confirm the statement that the disagreeable odor can be removed, except Pelouze possesses a means not published by him. Even when carefully avoiding an excess of sodium sulphide and expelling the sulphuretted hydrogen by continued heating, a disagreeable odor remained behind which could not be removed.

J. Laurent raises the following objection to Pelouze's method, which deserves attention. Two factories in Marseilles manufacture daily 44,000 pounds of soap, and if they should adopt this new method, 500 cubic meters (17658.29 cubic feet) of sulphuretted hydrogen would be daily developed, the injurious influence of which upon the health of the workmen and the hygienic condition of Marseilles would be incalculable.

*Direct saponification of oil fruits.*—Liebreich has patented a process for the direct saponification of copra.\* The copra is comminuted, then saponified with caustic soda lye, and the soap paste separated from the cellulose by means of a centrifugal machine. By salting out the paste a grained soap is obtained which is so hard that it cannot be cut by any of the usual cutting machines.

Soaps made of cocoanut oil are difficult to salt out, and when finally separated by the use of too much salt, inclose a considerable quantity of the latter. Soaps formerly prepared, according to Liebreich's process, by the Manufactory of Chemical Products at Charlottenburg, from pure copra or from copra and some olein, showed the above defect of containing a considerable quantity of salt, and made the hands rough.

Liebreich claims as an advantage of his process that it is the only sure method by which a neutral soap can technically be produced. It is difficult to comprehend why such should be the case, as there can be no doubt that a neutral soap can be prepared by the usual methods of salting out and grinding.

*Preparation of soap by the conversion of calcium oleate with alkaline carbonate.*—As far back as 1873, Tardoni entered in England a patent for the conversion of lime soap into soda soap by treatment with sodium carbonate, but the process appears to have never been carried on on a large scale. The idea has recently been again taken up by Krebitz,† his method being as follows:

\* The dried meat of the cocoanut.

† German patent No. 155108 was granted to Peter Krebitz for the preparation of an insoluble soap, the constitution of which renders possible the ready separation of glycerin.

The fats are melted, heated to 212° F., and saponified with lime. For 100 lbs. of fat about 14 lbs., more or less, of lime, according to its quality, are required. The lime is slaked with water and gradually stirred into the fat previously heated to 212° F. When all the lime has been introduced heating is discontinued, the kettle tightly covered and allowed to stand quietly over night. Spontaneous heating takes place, and the next morning the lime soap lies finished in the kettle. It is taken out, ground and sieved, when it is brought into a filtering tank and washed twice with hot water to obtain the glycerin. The glycerin-liquor thus obtained has a density of 5 to 6° B. The lime-soap is allowed to drain off, then returned to the saponifying kettle, and for every 100 lbs. of fat used 21 lbs. of soda, previously dissolved in water to about 30° B., are added. Conversion is effected with the assistance of steam or over an open fire. The lime combines with the carbonic acid of the soda to insoluble carbonate of lime which settles on the bottom, while the fatty acid forms soap with the soda. To those who are inexperienced, it is recommended to use a small quantity of caustic soda, though it is not necessary. The soap is like every other soap ground with salt, and eventually separated. The lime must be thoroughly washed to obtain all the soap. The resulting soaps are light in color; they contain traces of lime. The glycerin of course contains fewer foreign constituents than are contained in the spent lyes, but is more impure than that obtained by autoclave-saponification. The process was formerly not available for soft soap, but means have now been found to make it so.

Opinions regarding the value of Krebitz's process vary very much. Its drawbacks are that it is troublesome, further the difficulty of inducing complete saponification of the fat with lime without the use of an excess of the latter, of obtaining all the glycerin from the lime soap, of attaining complete conversion of the lime soap with alkaline carbonates, and preventing soap or fat from remaining in the carbonate of lime, or lime in the soap. Thus satisfactory results can only be arrived

at by carrying on the operation with the greatest care and attention. Another drawback is the large residue of carbonate of lime. Krebitz saponifies with 14 per cent. of lime, and thus returns to the oldest method of lime saponification, in which 14 to 15 per cent. of lime was used, while theoretically not even 10 per cent. is required. While in Krebitz's process there is no expense for sulphuric acid for this large quantity of lime, it has still the disadvantage that the larger the insoluble residue is the greater the danger of soap or fat remaining in it. Besides many plants would find it difficult and expensive to get rid of this residue, although others might be able to sell it, mixed with ashes, as a fertilizer, or thoroughly compounded with water, discharge it into a stream.

*Method for the preparation of soaps of a light color.*—Carl Dreymann \* prepares from impure, and also badly-smelling, fats and oils, even with the use of a very large percentage of rosin, firm and odorless soaps of a light color, by washing the finished soaps which contain the impurities with a 3 to 20 per cent. soap solution. By continuous crutching of the soap in the washing fluid, the impurities settle on the bottom of the kettle, the results being complete purification, and also removal of the bad odor. To remove the excess of water the soft-soap mass is then, according to its kind, boiled with brine of 9 to 20° B. The brine must be of sufficient strength to fix enough water so that the soap after salting-out only shows a yield of 145 to 148 per cent. Even in this state the soap is too soft and watery, and has to be hardened in a separate kettle by crutching in 2 to 3 per cent. of brine of 2 to 10° B.: the less water the soap contains the weaker the brine should be. In place of the latter, solutions of sodium sulphate, potassium sulphate, potassium chloride and silicate of soda, or soluble glass, or also soap solution may be used. It is claimed that in this manner a uniformly firm soap with a dry feel and of light color, without the stickiness of rosin, is obtained.

\* English patent No. 3973, 1903.

*Saponification of fatty acids according to the ammonia-soap process.*—A transposition similar to that of ammonium bicarbonate and common salt to sodium bicarbonate and ammonium chloride—the ammonia-soda process—can also be entered into by the ammonia salt of the higher fatty acids with sodium chloride, soda-soap and ammonium chloride being formed—ammonia-soap process.

The utilization of this chemical process was first proposed, in 1859, by J. C. Leucks, who recommended that, where ammonia could be obtained at a low price, it might be used for saponification, especially of olein, and the resulting ammonia-soap be decomposed by common salt into soda soap and ammonium chloride.

Independent of this the English chemist, Whitelaw, described, in 1876, in the "Chemical News," a method for the preparation of soap direct from common salt, and, according to him, 100 parts fatty acid, 15 to 20 parts ammonia, 20 to 30 parts common salt and 200 to 300 parts water, were to be taken for the purpose. The conversion was to be effected with the assistance of heat.

The conversion of fatty acid according to the ammonia-soap process was also the object of two German patents of 1888 and 1893.\*

Stiepel has recently given a more detailed description of the ammonia-soap process as follows: As initial material, which is to be converted into ammonia soap, free fatty acid is only available. The ammonia soap could, to be sure, be prepared direct from neutral fat by saponification with ammonia, but the process is not rational as contrasted with the method referred to, which is divided into two stages: 1. The conversion of the fats into fatty acids according to one of the known methods of saponification, and 2, the preparation of the ammonia-soap from fatty acid and ammonia.

The preparation of the latter succeeds very readily by

\* German patents Nos. 43340 and 72921.

bringing the liquid fatty acid into ammonia water, the ammonia soap forming thereby, according to the kind of fatty acid, a more or less viscid, jelly-like mass. By adding, whilst constantly stirring, common salt solution to this ammonia-soap, soda soap separates upon a watery spent lye. According to Stiepel, a saponification of 95 to 100 per cent. is effected when working in the cold. The ammonium chloride is said to be regained by repeated washing of the soap. The soap is obtained in small grains, which by suitable machinery have to be brought into salable shape.

*Saponification of fatty acid with silicate of soda or soluble glass.*  
—The earliest reference to the saponification of fatty acids by means of silicate of soda is found in an English patent,\* granted in 1855, to W. Gossage, for “a method of preparing certain kinds of soap.” The object of the invention was the preparation of soap from rosin and fatty acids by saponifying them with silicate of soda solution, or from neutral fats by mixing them with calculated quantities of silicate of soda, and boiling. Stiepel tested the latter proposition and found that neutral fats cannot be successfully saponified to any extent in a simple manner by silicate of soda solutions; on the contrary, only free fatty acids can be converted into soap by silicate of soda, the silicic acid being separated. A subsequent saponification of the neutral fat after the saponification with silicate of soda, by means of caustic alkali with the use of, for instance, 85 per cent. fatty acid, as is possible in the soda saponification is, in this case, not practicable for the following reason: In soda saponification, just as in silicate of soda saponification, a weaker acid is expelled by the fatty acid, carbonic acid in the first case and silicic acid in the latter. The fundamental difference between the two kinds of saponification consists in that in the first the separated acid—carbonic acid—escapes as a gas from the soap mass, while the silicic acid remains in it as free acid. Now by adding caustic alkali to this soap mass, it

\* English patent No. 908.

continues, in the soda saponification, to exist as free alkali ; while in the silicate of soda saponification it again combines with the silicic acid to silicate of soda. The caustic alkali can therefore become effective in the saponification only after all the silicic acid has been reconverted into silicate of soda. For this reason the preparation of such silicate of soda soaps can be of advantage only when working with about 100 per cent. fatty acids.

It would seem that in the preparation of firm soda soaps, which should not contain more than a certain percentage, the use of silicate of soda by itself is not applicable since silicate of soda of 35° B. contains merely 8 per cent. of saponifiable alkali. Hence 100 parts of fatty acid with a saponification-number of about 200 would require about 180 parts of silicate of soda for complete saponification, and the result would be a yield of 280. However, such product would turn out too soft. For the preparation of firm soda soaps a mixture of caustic soda and silicate of soda will therefore have to be employed. As stock for such a soap may serve : 100 lbs. tallow fatty acid and 40 lbs. rosin, saponified with 82.5 lbs. silicate of soda in which 18.5 lbs. solid caustic soda have previously been dissolved. The fat is heated to the melting point and silicate of soda solution at 176° F. is, whilst constantly stirring, added as rapidly as possible, the temperature rising thereby considerably. The mass is stirred until intimate mixture and saponification have been attained and a perfectly uniform mass has been produced. The mass while semi-liquid is brought into the frame to cool. Further filling with silicate of soda is not excluded.

The preparation of potash soaps may be effected in the same manner with the use of a considerably larger percentage of silicate of soda.

While this method of preparing hard soaps possesses pecuniarily no material advantage over the customary process, the products obtained might, however, prove superior to other soaps in cases where a large amount of dirt has to be re-

moved. The silicic acid separated during the saponification process is, to be sure, present in a finely divided state. It becomes, however, effective as a rubbing agent in using the soap whereby the cleansing action is considerably augmented. A cheap laundry soap which thoroughly cleanses in this manner without the presence of strong caustic alkalies, would be in demand for many purposes, especially for washing working mens' clothes.

*Means for correcting the alkaline action of soap.* It is a well known fact that many a sensitive skin cannot stand washing even with perfectly neutral soap. The skin becomes dry and there is a peculiar burning sensation which may be relieved by the application of cold cream or dilute glycerin. The explanation of this effect of the soap is found in the fact that the alkali, which splits off when the soap is dissolved in water, withdraws oil from the skin. For obviating the alkaline effect of neutral, as well as of slightly alkaline, soaps, various additions to the soap have been recommended. For medicated soaps Unna proposed to super-fatten them, and for this purpose gave the following formula: Best beef tallow 16 parts, olive oil 2, soda lye of 38° B. 6, potash lye of 38° B. 3. He recommends this soap not only for skin diseases, but also to physicians who have to wash their hands frequently in the course of a day. Since soaps which contain free fat turn readily rancid, Dr. Ewald Geissler has recommended the use of fatty acid in place of neutral fat. It has recently been sought to secure the same object, especially with toilet soaps, by an addition of lanolin. However, as under ordinary conditions lanolin is non-saponifiable, its action can only be a mechanical one, the same being very likely the case of neutral fat in over-fatted soaps.

Since white of egg (albumen) possesses the property of forming with alkalies a non-caustic alkaline albuminate, soaps with white of egg have repeatedly been prepared and some patents \*

\* German patents Nos. 112,456, 122,354 and 134,933.

for methods of preparing them have quite recently been granted. On account of its high price white of egg can only be used to a limited extent, and attention has been directed towards other varieties of albumen, such as serum albumin or blood albumen and the casein of milk, the latter being preferable, as its color is almost white, while that of the former is yellow. Casein combines with free alkali as well as with carbonates to an albuminate which in watery solution is also not subject to hydrolysis.

According to Dr. Karl Braun, soaps have recently been recommended that have been rendered neutral by the addition of malt extract and also of malt sugar. The free alkali forms with the sugar a sodium saccharate. Soaps with such an addition are claimed to possess great lathering power. Dr. Braun also considers mucilage or gum as suitable for fixing the free alkali in toilet soaps. He also recommends borax for the same purpose. By dissolving borax in water, decomposition takes place, according to Dr. Braun, into sodium metaborate and free boric acid,  $\text{Na}_2\text{B}_4\text{O}_7 + 3\text{H}_2\text{O} = 2\text{H}_2\text{BO}_3 + 2\text{NaBO}_2$ . The boric acid fixes the free alkali to a borate.

Wilhelm Rödiger has patented a method for freeing soaps from all adhering caustic soda by the introduction of bicarbonates, sodium bicarbonate being added to the soaps after fitting.\* The sodium bicarbonate on coming in contact with caustic alkalies yields one-half its carbonic acid and forms either with the free alkalies alkaline carbonate or, as the patentee has patented the process for paste soaps, with the carbonates a sesqui salt. The toilet and shaving soaps prepared with the addition of sodium bicarbonate are brought into commerce by Kluge & Co., of Magdeburg, Germany, under the name of "patented normal soaps."

\* German patent No. 27,325.

## CHAPTER XI.

### HARD SOAPS.

#### I. GRAINED SOAPS.

##### a. *Grained Soap upon Spent Lye.*

THE boiling of grained soap upon sub-lye or spent lye may be divided into three principal operations: Preparatory boiling or pasting; salting-out, or, as it is sometimes called, graining; and clear boiling or boiling on strength. The object of the first operation is the saponification of the fats, that of the second the separation of the soap from the glycerin and the superfluous water, and that of the third to complete saponification and to ensure that the soap is quite free from unsaponified fat. The grained soaps are generally named after the principal fat used in their preparation, as, for instance, tallow grained soap, palm oil grained soap, olein grained soap, rosin grained soap, etc.

*Old German grained soap.*—Prior to the introduction of soda, potash was exclusively used for the saponification of fats, and, if grained soap was to be prepared, the soft soap resulting from the saponification of the fats was decomposed by common salt. The alkali required was originally obtained from wood ashes by intimately mixing the latter with slaked lime and adding a certain quantity of water to the mass; the resulting caustic lye being used for boiling. Later on the use of potash was introduced in many soap-boiling plants, as it afforded a cleanly, convenient and sure means of working. Although at the present time but little grained soap is made with the assistance of potash, a description of the process is here given, as it is of historical and theoretical interest.

The tallow is brought into the kettle, the lower portion of which consisted generally of cast iron and the upper of wood, lined with roofing tiles, as a rule laid in cement. A few pots full of lye of 8° Bé. are then added and the fire started. As soon as the tallow is melted and the lye heated a milky fluid (emulsion) is at once formed by crutching. After further heating a clear soap solution mixed with fat particles appears, and combination between fat and alkali soon sets in. That combination has taken place is recognized by the yellow-brown mass boiling quietly while gradually rising. A sample adhering to a spatula dipped into the mass has a jelly-like, gray-white appearance, and no lye separates. However, the mass in the kettle is not yet soap, as it still contains too much unsaponified fat, and if evaporated in this state would result in a thick, gray grease, which could again be dissolved by the addition of lye. Hence, while the mass is quietly boiling in the kettle, more lye, generally two-thirds of 8° B. and one-third of 20° B. is added. The addition of lye is continued until a viscid, homogeneous, clear mass is in the kettle, which runs off the spatula in cohesive, long, transparent strings; or, as it is called, *the soap boils in paste*.

From the condition of the paste it is seen whether complete saponification has taken place and whether the proportion of alkali to fatty acids has been correct. If some of the soap be dropped upon a glass-plate and this sample for some time remains clear and becomes turbid only on cooling, the paste is of the proper quality; if, on the other hand, the sample becomes in a short time turbid, such turbidity is due either to unsaponified fat distributed in small particles in the paste, or to the addition of too much lye and the consequent separation of solid soap. Unsaponified fat is present if a gray border appears immediately on the edge of the sample, while in the presence of an excess of alkali the sample becomes rapidly coated with a whitish film. Such defects are readily corrected by the addition of lye or tallow. When the paste is sufficiently clear, and a sample brought in contact with the

tongue causes a slight burning sensation, or shows, as it is technically termed, a slight *touch*, it is vigorously boiled in order to deprive it of a portion of the superfluous water, until it runs in threads from the spatula, which is termed *spinning of the soap*. As soon as this moment arrives, the separation of the soap from its aqueous solution by means of common salt is commenced, which at the same time effects the transformation of the potash soap into suda soap.

The salt is added gradually and in small portions, and after each addition the paste is allowed to boil for a short time in order to enable the operator to watch the full effect of the salt, which does not show itself at once. The first addition of salt effects, as a rule, a liquefaction of the paste. After further additions, the soap coagulates and finally comes as a thick mass to the surface. The separation is due to the soap being insoluble in salt solution and the salt withdrawing water from the soap. The fluid separated from the soap, which is called sub-lye or spent lye, contains, besides the common salt added, the potassium chloride formed by the decomposition and the glycerin formed by the saponification.

Great attention was formerly paid to the operation of salting out, since with an insufficient addition of common salt a portion of the soap remains dissolved in the spent lye, and with too much salt the soap separates too quickly, so that there is danger of small lumps being formed which combine with difficulty and hence absorb spent lye. Soap properly salted out must, when taken hot upon the spatula, adhere to it in soft flakes, boil into *laminæ*, and a sample, on being pressed with the thumb in the palm of the hand, must not smear, but peel off as a solid, dry chip, or, as it is technically called, *have pressure*. The spent lye, when tested, should not have a pungent, but a saltish-sweetish taste.

When salting-out is finished, the fire is withdrawn and the spent lye, when thoroughly settled, is drawn off through a stop-cock near the bottom of the kettle or pumped out; or the entire contents of the kettle are transferred to the cooling tank

and allowed to rest for the spent lye to settle. A quantity of the weakest lye is then brought into the empty kettle, and the soap, being carefully removed from the spent lye in the cooling vat, is added and dissolved to a clear paste, which is again carefully salted out. But little salt is required for this operation, as it only serves for the absorption of water. The next operation, *clear boiling* of the soap, is now proceeded with.

The object of clear boiling is to withdraw superfluous water from the soap, to saponify the last remnants of fat, and to form the soap into a solid mass free from froth. This operation was generally executed by allowing the soap to boil up high with a quiet, uniform fire, keeping the paddle always ready to prevent boiling over by beating the soap, or, as it is called, *checking* or *curbing* the soap. The soap at the beginning not sufficiently separated from the spent lye soon seethes up in small bubbles. By continued boiling, with the kettle usually half-covered, water is gradually evaporated, the salty lye becomes more and more concentrated, and by withdrawing more and more water from the soap the latter becomes more contracted and viscid. Large laminae form on the surface, and the steam developed on the bottom of the kettle escapes with great noise through the thickening soap. The froth now full of large bubbles subsides gradually, the soap forms a homogenous mass, and no more water being withdrawn from it, clear boiling is finished. On repeating the test with the thumb the soap can be rubbed to a dry, pliant chip. After withdrawing the fire, the soap is allowed to rest a few hours for the spent lye to settle thoroughly, and is then brought into the frame. When such a clear-boiled viscid soap is brought into the frame a slight mottle is readily produced in it by adhering impurities. As mottled soap was formerly considered especially good, bole elutriated in lye, pyrolusite, etc., were frequently mixed with the soap before bringing it into the frame, in order to make the mottling more pronounced. If, on the other hand, smooth soap was desired, the thick grain was ground with hot water or weak lye, which rendered the

soap more liquid and the adhering impurities passed into the spent lye. By grinding, the soap absorbs some water and the yield is by this manipulation somewhat increased. One hundred pounds of tallow give about 150 pounds of unground grained soap, while by grinding the yield may be brought up to 156 pounds.

Framed soap prepared with potash lyes are more delicate and smoother than those made directly with soda lye, but they are far more expensive, potash not only being considerably dearer than soda of the same grade, but a larger quantity of it is also required. Suppose both substances were chemically pure, then 100 pounds of tallow would require for saponification 28 pounds of chemically pure potassium carbonate, or 21 pounds of chemically pure sodium carbonate. From what has been said it will readily be understood why the use of potash lyes in the manufacture of hard soaps is very limited, it being almost restricted to the preparation of shaving soaps.

*Tallow grained soap.*—This soap is still very much liked in the household on account of its great economy in washing, and in the textile industry by reason of its great unctuousness and other good qualities. Besides, tallow is the best raw material for the manufacture of grained soap and gives the largest yield, 100 pounds producing 150 to 160 pounds of soap according to whether the latter is more or less ground.

The lye required for the manufacture of this soap is prepared from high-grade soda ash set with 50 to 60 per cent. of lime, or by dissolving high-grade caustic soda in water. The use of the latter has the advantage that in salting-out no sodium carbonate—more or less of which is always contained in lyes prepared from soda ash—is lost. Sodium carbonate does not contribute to saponification and, being only mechanically mixed with the soap and deposited between the soap atoms, renders the soap more fluid. When the soap is salted-out the sodium carbonate in the paste not being chemically fixed in the soap, is mostly carried into the spent lye. The preparation of tallow grained soap by means of soda lye is much the

same as that of old-fashioned grained soap with the use of potash lye. However, the soap is now generally boiled upon one water, except where the impurity of the tallow renders a more frequent renewal of the lye necessary.

The boiling process is generally carried on as follows: The tallow is brought into the kettle, and as it can only be completely saponified with weaker and more caustic lye, tank-lye of  $8^{\circ}$  to  $10^{\circ}$  B. (about one-quarter of the entire quantity required for saponification) is added. With a slow fire, the lye and tallow immediately form an emulsion and combine to a paste shortly after the boiling point is reached. This combination, and consequent complete fixation of the alkali by the fatty acids, are recognized by a sample brought in contact with the tongue showing no longer a biting, but a more sweetish, taste. However, if the mass still shows some sharpness, the complete fixation of the alkali must be effected by continuing the boiling.

As tallow, compared with most other fats, only gradually saponifies, a few more pottuls of lye are added after combination has been effected. In the same manner more lye of  $12^{\circ}$  to  $13^{\circ}$  B. is gradually added to the soap mass and boiling continued until there is clear, viscid mass—the soap paste—in the kettle.

If it should happen that by adding the lye too rapidly a paste is obtained which, though showing sharpness when brought in contact with the tongue, has a turbid and dull appearance, some very weak lye has to be added, while boiling slowly, when the sharpness will disappear and the paste become clearer; any fitting by lye which may still be required will then also become evident. An addition of water or of weak lye is also of advantage when by the use of too strong a lye ( $18^{\circ}$  or  $20^{\circ}$  B.) the combination has been destroyed so that the lye runs off separately from the spatula, and if necessary the fire may also be withdrawn from under the kettle and the soap allowed to rest for a few hours, when it will again come by itself into good combination. The use of boiling-lyes too

low in lime, *i. e.*, such as still contain much alkaline carbonate, can only be injurious, since they not only enter with difficulty into combination with tallow, but the large quantity of sodium carbonate always contained in them is liberated by salting-out and passes into the spent lye.

The saponification of the tallow may be considered complete when a sample of the paste brought upon a glass plate shows immediately a narrow whitish border (lye-rings), remains clear for some time, becomes turbid only on cooling, and when brought in contact with the tongue gives a perceptible burning sensation (touch).

To withdraw a portion of the superfluous water the soap is now vigorously boiled until it runs in cohesive threads from the spatula, or, as it is called, *spins*. When this is the case its separation from the watery solution is proceeded with, which, as a rule, is effected by means of common salt. For this purpose salt in small portions is added to the soap paste, when it soon becomes more liquid. By the gradual addition of more salt the separation of the soap from the watery solution (spent lye) is then effected.

Salting out is best effected by keeping the soap at a gentle boil. Soap properly salted out should boil into laminae, and when taken upon the spatula, show white flakes and have pressure. The spent lye should run off clear from the spatula and should not have a pungent, but rather a more salty, taste. It may be mentioned that for salting out soap paste prepared with soda lye, less salt is required than for that with potash lye alone, since in the latter case a certain quantity of salt is needed for the decomposition of the potash soap.

Salting out being completed, the fire is removed for the time being to allow the soap to quiet down. A portion of the spent lye is then pumped off and the operation of clear boiling commenced, which is effected in the same manner as described for old German grained soap.

Many manufacturers prepare their grained soap without boiling it clear upon spent lye. The soap paste is only boiled

clear and free from froth, and then carefully separated with salt or strong brine so that no froth is formed, whereby a grain free from froth is obtained in the kettle. Grained soap thus prepared gives a greater yield, but remains always readily soluble, and when cut into bars frequently dries crooked. Such soaps can, however, be still more quickly and better prepared by means of steam. By the violent motion of the mass, produced by the admission of steam under strong pressure, the tallow is brought into more intimate contact with the lye and saponification is more readily effected, so that a large quantity of tallow is in a short time converted into a clear paste free from froth.

For the preparation of grained tallow soap with caustic soda lyes the process of boiling is the same as above. For pasting, caustic soda lye of 10° to 14° B. is used. If in working old, somewhat rancid tallow, which contains, as is well known, more free fatty acids, the soap is rendered too caustic, which is indicated by thick and viscid boiling, some brine of 20° B. is carefully added to remove this viscosity, or some soda solution where brine may be too strong and exert an injurious effect upon the combination. By boiling old, rancid fats upon pure caustic soda lyes, defective grained soaps are obtained, which is, however, also the case when fresh fats are boiled with lyes containing much alkaline carbonate.

*Mottling grained tallow soap.*—When the operation of clear boiling is finished, the fire is drawn and the spent lye allowed to settle. If the soap is to be mottled the grain is brought into the frame or first rendered somewhat more liquid by crutching in some hot water or weak lye, care being taken not to work in any spent lye. When the soap has been thoroughly crutched, the filled frame is covered with boards to permit the soap to become thoroughly heated, and allowed to stand quietly. By the soap becoming heated crystallization takes place and the *grain and mottle* are formed. The grain is the crystalline portion of the soap and incloses the non-crystalline (the mottle), all the impurities and coloring matter from the tallow, soda,

etc., contained in the soap passing into the mottle. Such soap when cut shows a more or less mottled appearance. To make the mottle more pronounced, the grain is frequently first colored with bole elutriated in lye, pyrolusite, Frankfort-black, ultramarine, etc. The coloring matter being mechanically mixed with the soap, deposits itself in the mottle.

The mottle in grained soaps is allowed to form either by itself or, after the process of its formation is complete in the soap, is drawn into a different design by artificial manipulation, the design resembling either sheaves closely arranged alongside each other or flowers. The process of producing sheaves is as follows:

When the soap in the frame has become sufficiently heated and set, which is recognized by the steam penetrating through the cracks between the boards, and shows the requisite fluidity, which otherwise has to be effected by crutching in some hot water, or weak lye, or by adding several pounds of cocoanut oil, the process of producing sheaves is commenced after allowing the soap to somewhat cool off.

A round iron rod, provided on top with a handle and below with a small button, is pushed into the soap down to the bottom. With this rod straight lines, as close to each other as possible, are first drawn the length of the frame and then the width. The closer and more uniform these lines are drawn the more beautiful the sheaves will be. Flowers are produced in the same manner; flower-like designs being formed by skilful manipulation and twisting of the iron rod.

Simple mottling, or sheaves and flowers, can, of course, be only produced when the soap is sufficiently hot, crystallization not taking place in soap brought too cold into the frame.

Smooth white curd soap is obtained either by crutching the soap in the frame, of course without the addition of coloring matter, until it is observed to congeal, whereby crystallization is prevented and the small quantities of impurities uniformly distributed, or the thick grain in the kettle is sufficiently ground.

*Grinding of soap.*—The object of this operation is (1) to prevent the formation of mottle, (2) to purify the soap if necessary, and (3) to increase the yield by the absorption of water. Grinding is effected by two different methods, either "*from above*" or "*from below*," but is always carried on with water or weak lye.

Grinding from above is generally executed by crutching into the thick-grained soap in the kettle enough hot water or weak lye to sufficiently dissolve and liquefy it. During this manipulation soap which is too sharp, *i. e.*, contains an excess of alkali, may, if necessary, be somewhat improved by grinding with water, by which the alkali is withdrawn into the spent lye, or some causticity may be imparted to the soap by grinding with strong lye. Soap of proper constitution is generally ground with weak lye ( $3^{\circ}$  to  $4^{\circ}$  B.), preferably potash lye. When grinding is finished the kettle is covered, and the soap allowed to stand a few hours for the impurities to pass into the spent lye.

Grinding from below is effected by pumping out or drawing off the spent lye, then bringing into the kettle the required quantity of weak lye or water with an addition of salt to prevent the complete formation of paste, and effecting solution and liquefaction of the soap by vigorous crutching or gentle boiling. This operation is only required with the use of very impure materials, and is but little practiced at the present time.

*Utilization of spent lye.* The spent lye from soaps contains common salt, the excess of alkalies used, the glycerin contained in the fat, frequently more or less soap, and the impurities from the fats and the lye. Though it contains valuable substances it is seldom utilized by soap boilers and generally allowed to run off, and where facilities for carrying it off are limited, frequently causes great inconvenience. Usually an attempt is made to utilize the alkali contained in it by adding fat, and boiling. The lyes which finally remain are seldom clear and limpid, but generally thick and congeal to a jelly-like

mass. The soap-boiler who is forced to allow such lyes to run off hot in order to obtain room for a new boiling loses many pounds of soap contained in them.

In many factories an attempt is made to utilize the spent lye by evaporating it and selling it as so-called "fulling extract." Evaporation is generally effected over an open fire, though indirect steam is preferable. With an open fire constant stirring is absolutely necessary to keep the bottom of the kettle free, which otherwise would suffer injury and soon wear out. Evaporation is continued until a sample congeals to a jelly interspersed with small crystals of soda and salt. Deep soap-kettles are not well adapted for the operation, the evaporating surface being too small and stirring difficult and almost impossible the moment the separation of crystals commences. Shallow pans can be highly recommended for the purpose, though they take up much space, of which there is seldom an abundance in soap-factories. In many factories the operation is carried on by evaporating the lye in a soap-kettle to the commencement of the separation of crystals and finishing in shallow pans.

Independent of the profit gained by selling the fulling extract, this evaporation of the lyes has the further advantage of drawing attention to the immense quantity of soap contained in them. This content of soap varies with different fats, and, besides, depends on the manner of salting out, the spent lye containing more soap if salting out has been effected by an excess of lye than by common salt. However, not every thick, jelly-like spent lye contains larger quantities of soap, bone fat, for instance, yielding frequently such thick spent lye, this being, however, due more to glue than to soap. The jelly-like spent lye from sulphur olive oil is due to vegetable albumen.

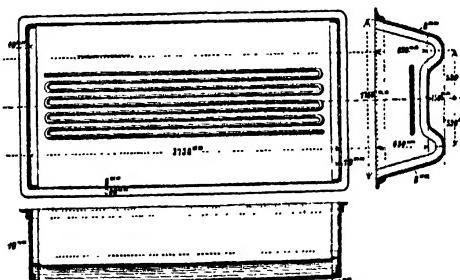
When the spent lye has been boiling for some time a froth forms on the sides; this is removed and added in boiling rosin grained soap. Spent lyes from rosin grained soaps are difficult to evaporate; they foam at first very much and have to be curbed by stirring. To lyes which contain very little alkali,

which is readily recognized towards the end of evaporation, a few pounds of soda are added.

The finished fulling extract is generally of a dark brown color. It is drawn off into old iron vessels, kettles, lye-kettles, etc., to cool. It remains in these receptacles until an order for the product is received, when it is filled into barrels, petroleum barrels being best for the purpose.

It has previously been mentioned that on account of the great content of common salt the recovery of glycerin from the spent lye is a difficult matter. On treating glycerin with animal charcoal only a very small portion of the salt is removed, and a not inconsiderable quantity of it is even carried over in distilling with superheated steam. These drawbacks for a long time prevented the utilization of spent lyes for the

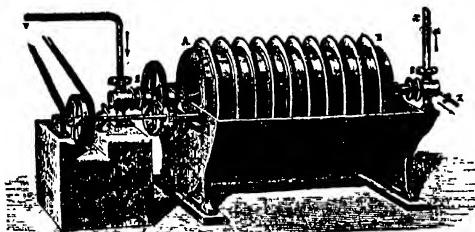
FIG. 33.



recovery of glycerin. Only when crude glycerin became scarce and its price had risen remarkably, more attention was paid to its recovery from spent lyes. Many patents were taken out for the purpose, but it would lead too far to discuss them here. In all methods for the recovery of glycerin from spent lye it is necessary to first evaporate the latter in order to free it as much as possible from salts. For this purpose it is best to use a shallow pan, such as shown in Fig. 33, which allows of a convenient removal of the salts, or Leon Droux's apparatus with revolving cylinder, shown in Fig. 34. It consists

of the metal cylinder *A B*, with the hollow axles *S S*, through which the steam is admitted and later on discharged. The heating surface of the cylinder is considerably augmented by a number of ribs on its circumference. The cylinder dips into the fluid to be evaporated. By a devise, as seen in the illustration, the cylinder is slowly revolved, and with each revolution is covered with a thin layer of fluid which readily evaporates even at a comparatively low temperature. The condensed water runs off at *Z*. The salts form an abundant deposit on the surface of the cylinder, and can be readily

FIG. 34.



removed by means of a hammer, or by dipping into a less concentrated liquid.

The lye is evaporated to  $30^{\circ}$  B. and then allowed to rest for some time. After cooling a portion of the chlorine combinations and of the sulphates separates out; the liquid, however, retaining sufficient sulphur combinations to render the recovery of the glycerin impossible. The sulphur combinations are, therefore, oxidized by an addition of sulphuric acid and the introduction of hot air. The liquid is then again evaporated, this time to from  $35^{\circ}$  to  $36^{\circ}$  B., the product being a highly colored, syrupy liquid.

Instead of neutralizing the original lye with sulphuric acid, evaporation can first be proceeded with to a certain degree, this method offering the advantage that a considerable portion of the soda and common salt contained in the spent lye can be recovered.

With indirect steam the spent lyes can without difficulty be brought to a concentration of from 32° to 33° B. During evaporation salt is abundantly separated; it consists of a mixture of common salt and soda, three times as much of the former as of the latter, and after draining off can be again used for salting out.

Since, as previously mentioned, in boiling these solutions of glycerin, a portion of it is carried away by the escaping steam, losses in evaporating spent lyes are unavoidable.

The lye evaporated to 32° or 33° B. is neutralized with sulphuric acid, whereby large quantities of Glauber's salt are separated.

Since by reason of the development of the carbonic acid from lyes containing soda it is difficult to obtain an exactly neutral lye, it is best to add some sulphuric acid in excess and to neutralize the latter, when precipitation is finished, by the addition of lime. Whether sufficient sulphuric acid has been added is recognized by blue litmus-paper when dipped in the lye acquiring a brick-red color. The lye treated with sulphuric acid is allowed to stand quietly over night for the Glauber's salt to separate.

The clear lye when drawn from the Glauber's salt is again concentrated with indirect steam to 32° or 33° B. In cooling, crystals of sodium sulphate and common salt separate from the lye, and the latter is now ready for further utilization.

Generally speaking, it will be best for the soap manufacturer who wishes to utilize his spent lye for the recovery of glycerin to proceed only up to this point, or simply to evaporate the lye to 30° B., and leave the further treatment to the manufacturer of glycerin.

As the content of common salt is the chief hindrance to the recovery of glycerin from spent lyes, Jaffé and Darmstädter have recommended the use of Glauber's salt instead of common salt for salting out, as it can be almost completely removed by evaporating the lyes, and, besides, does not pass over with the vapors in distilling with superheated steam. It has been pre-

viously mentioned that other salts, and among them Glauber's salt, possess the property of separating the soap from the aqueous solutions, but in a less degree than common salt. The separation by means of Glauber's salt being not a complete one, the spent lyes contain larger or smaller quantities of soap, and the process has the further disadvantage of pale soaps frequently becoming dark on storing.

*Marseilles soap.* The product known as *Marseilles, Castile* or *Venetian* soap is a ground, grained soap prepared from low-grade olive oils which are not suitable for comestible purposes. Olive oils for the manufacture of soap, as found in commerce, vary very much. They may be more or less thinly fluid, then again very thick—the so-called olive oil foots—of a pale or greenish-yellow color, the latter passing into the darkest grass-green in what is known as sulphur oils. The resulting soaps also vary according to the consistency and coloration of the oils used in their preparation. There are, for instance, Marseilles soaps white as snow, yellowish-white to greenish-white and quite green, leaving out of consideration the grass-green sulphur-oil soaps, which are a variety by themselves. Soaps showing the least color are most highly valued and bring the best price. The soaps prepared from the more fluid oils possess the property of being very thinly fluid and keeping warm for days, even when previously boiled for a long time in grain. By much grinding they can be made almost as liquid as water, and kept in this state for days in the frame. Naturally such soaps must become very pure and neutral, there being plenty of time for the precipitation of all the impurities and the greater portion of the excess of alkali, as compared with the ordinary grained soaps made from solid fats, which as a rule harden on the edges the second day in the frames; with Marseilles soap this requires on an average eight days or longer.

The oils to be saponified have been so firmly fixed by custom that scarcely anybody would think of boiling Marseilles soap without olive oil. Good yields and, in most cases, quite

a white soap also, are obtained from olive oil foots, because mostly the portion of the oil containing stearin and palmitin settles by long storing of the oil on the bottom of the reservoir. Other oils or fats may, however, without detriment be substituted for a portion of the olive oil, a good quality of olein being primarily very suitable for this purpose, as soap prepared from it is in every respect equal to that from olive oil. Up to one-half the quantity of olive oil may also be replaced by peanut oil. Tallow is less suitable as a substitute, though fairly good results are obtained by the use of not more than one-quarter of it in the stock.

The use of cotton-seed oil as a partial substitute is on the other hand less advisable, since the results thus far obtained with it as an addition to white soaps can by no means be called satisfactory. On account of the depressed price of soap, the consumption of cotton-seed oil, for instance, for white grained soap, is at present very large, and the soaps prepared from it are, when fresh, excellent and without defect. However, the property of the oil to after-yellow in the soap when the latter is stored for some time cannot be overcome, notwithstanding previous careful bleaching and correct saponification. For this reason it is best to omit cotton-seed oil entirely from stock for Marseilles soap. Such soap lies often for months in stores, and while the at first somewhat greenish color may be expected to fade to some extent, it should not change to a yellow-mottled one.

When working with caustic soda the operation of boiling the soap is quite simple, no matter whether olive oil alone is used or in connection with the above-mentioned oils or fats. The strength of the lye to be used depends on the composition of the stock, and will be referred to later on, but the surest and most accurate work is done by calculating the caustic soda for a determined quantity of oil.

If caustic ammonia soda of 120 to 128 per cent. be first considered, we know that from 100 lbs. of it, 300 lbs. of lye of 38° B. are obtained, and that these 300 lbs. of lye are sufficient for the saponification of exactly 600 lbs. of oil or fat.

Hence with 1 lb. of caustic soda 6 lbs. of oil or fat are so far saponified that neither unsaponified fat nor an excess of alkali can be traced in the soap. One pound of fat requires for saponification  $\frac{1}{6}$  lb. of caustic soda, 100 lbs. of fat, therefore,  $100 \times \frac{1}{6} = 16\frac{2}{3}$  lbs. of caustic soda. It does not matter in how much water this caustic soda is dissolved, because the larger or smaller quantity of water determines only the strength of the lye and the content of water in the soap-paste prepared with this lye; the soap-paste acquires its normal content of water by the subsequent salting out to grain. Suppose, for instance, 1000 lbs. of stock are to be completely saponified, then the quantity of caustic soda required for saponification is found by multiplying 1000 by  $\frac{1}{6}$ :  $1000 \times \frac{1}{6} = 166\frac{2}{3}$  lbs.

Hence for the saponification of 1000 lbs. of fat  $166\frac{2}{3}$  lbs. of caustic soda will have to be used. By consequently converting this caustic soda by dissolving in water into, for instance, a lye of 24° B., complete saponification would result in treating with it the 1000 lbs. of fat referred to. There will be small differences in this calculation, but only in so far as the saponification value of the various fats and fatty acids is not alike; however, small corrections always suffice to get the right proportion. This calculation, of course, only applies when working with caustic soda; additions of alkaline carbonates change the proportion, but in this case such additions are not necessary.

The procedure of boiling the soaps will be explained by two examples. Suppose the following stock is to be used: Olive oil 500 lbs., tallow 250 lbs., peanut oil 250 lbs., caustic soda  $166\frac{2}{3}$  lbs. The soda is made into a lye of 24° B. However, the lye may also be divided and one-quarter of it reduced with water to 10° or 12° B.

Now bring the weighed fat into the kettle and add 100 to 150 lbs. of lye, it being most convenient to do this, if possible, the evening before boiling. The lye is thoroughly crutched together with the oil, without heating, and allowed to stand

quietly till the next morning, when the lye will be found completely saponified, the soap formed lying on the bottom of the kettle and the liquid oil, together with the tallow, floating over it. The fire is now slowly started until the entire mass is hot. When the soap lying on the bottom of the kettle commences to boil, which is recognized by ascending bubbles, 100 lbs. more of lye are added and crutched in, and firing is continued. With slow firing and crutching and the successive addition of lye all the oil soon comes into combination, which can, moreover, be much accelerated by the addition of a few potfuls of water, or, as above mentioned, by reducing a portion of the lye to 10° or 12° B., which of course has then to be worked first. Thickening or running together of the soap must be prevented by the addition of lye; in an emergency a potful of salt will do no harm. However, the best protection against thickening is to work the lye as strong as possible. It is also permissible to make from the start the lye in three different strengths, for instance, one-quarter of it 12° B., one-half 24° B., and the last quarter 30° B. Thickening of the soap can then be always guarded against by the lye of 30° B. When all the lye has finally been worked up, and no salt has been used, there will be in the kettle a nice, clear soap-paste which already shows good pressure between the fingers; it should not produce a biting sensation on the tongue. Should the latter, nevertheless, be the case, boiling is continued for half an hour, and this defect, if necessary, remedied by the addition of small quantities of fat. However, only a small correction will be required if fats free from dirt and water have been used, because the calculation of alkali was, so far as it can be made, correct. The paste is then sufficiently salted out to obtain a nice grain, and tested as to its being perfectly neutral, which must always be the case with Marseilles soap.

For this purpose the method with phenolphthalein, previously referred to, is preferable, it being the most reliable known. Procure from a chemical supply house phenolphthalein solution and a bottle of 50 per cent. alcohol. Take a

sample of the soap from the kettle and, when cold, cut it up into fine shavings. Bring the shavings into a small test-flask, pour alcohol over them, and shake until they are dissolved. Drop a small quantity of phenolphthalein solution into the solution and note the result of the test. If the soap solution acquires a red color, the soap still contains an excess of alkali, and oil or fat has to be added to the soap in the kettle and boiling continued until a sample is no longer colored red, *i. e.*, until the soap no longer reacts. The kettle is then well covered and the soap allowed to stand till the next day to settle thoroughly. When it lies thinly fluid and ground in the kettle it is brought into frames more shallow than high.

On account of the special importance of the neutrality of Marseilles soap the test with phenolphthalein solution may again be referred to. Do not be misled if the solution acquires continuously a red color, notwithstanding repeated additions of fat. It might be supposed that the soap becomes too weak, especially if when tasted there is not the slightest biting sensation on the tongue. The test by the tongue is, in this case, not reliable and sufficiently sensitive; phenolphthalein solution, on the other hand, indicates the slightest trace of an excess of alkali, a deep red color being produced in the presence of great alkalinity. A scanty excess finally produces only a slight rose-red coloration. Even these slight traces of alkali must be removed, for the chemist examining such soap would say "neutral to slightly alkaline." Hence there should be no anxiety about the soap becoming too weak. The soap solution should appear white-yellowish, opalescent. Some care is only required with the last additions of fat, when the solution shows the rose-red coloration. If, on the other hand, the soap is to be boiled with tank lyes, the only way is to boil the oils to paste, salt out and clear-boil the grain.

The soap, as previously mentioned, remains liquid for a long time in tall frames, and must be frequently crutched. At a certain stage of cooling it should even be beaten with a heavy instrument, as otherwise the upper layer will bulge too much.

A good quality of olein, as previously mentioned, may also to a certain extent be used as a substitute for olive oil. A formula for this purpose is as follows: Olive oil 500 lbs., peanut oil 250 lbs., olein 250 lbs., caustic soda 166 $\frac{2}{3}$  lbs.

In working with olein, and this applies also to other soaps, one-half of the lye required is at once brought into the kettle and, when heated, the olein is introduced. When the latter is completely saponified and lies as a compact grain in the kettle, the other oils are gradually brought in. The soap will then by itself pass into paste, and boiling is finished as above described. Care must be taken not to attempt neutralization of an excess of alkali with olein, because the latter, immediately after having been added, comes to the surface of the soap in a finely divided, semi-saponified, spongy state, and boiling for hours is frequently required to combine these spongy lumps with the other soap. The grain when clear-boiled is brought upon fresh brine of 15° or 16° B. until all the excess of alkali has been washed out, which under certain conditions may require three to four days, while when boiling with caustic soda the object can be conveniently attained in one day. Purification of the oils, which is eventually to be effected by repeatedly washing the soap-grain with brine, can be more conveniently done by bringing the oils and fat, previous to boiling them, upon sufficient water, adding some salt, and letting them boil up several times. The oil when carefully removed from the water will be sufficiently pure. The purification of very slimy or dirty oils can, moreover, be better effected by the addition of a small quantity of lye.

*Palm-oil grained soap.* On account of its large content of palmitin and its being readily saponified, palm oil by itself is frequently used for grained soaps, but more generally in combination with other fats, such as tallow, bone fat, olein, etc., the resulting soaps being much liked in the various branches of the textile industry, as well as in the household. Comparatively little palm oil is used in the crude state, it being generally first subjected to a bleaching process to remove coloring matter and other impurities.

Pure palm-oil grained soap is prepared as follows: A determined quantity of bleached palm oil, together with a portion of the tank lye of 14° B. required (about 550 lbs. of lye to 1000 lbs. of oil), are brought into the kettle. With a slow fire and crutching, an emulsion is soon formed, and when boiling commences, an intimate combination between fat and lye takes place. When this is the case more lye of 14° B. is gradually added, whereby the mass is by degrees converted into paste, which, after continued boiling and the further addition of lye of 14° B., becomes thicker and shows a clear uniform appearance. When a sample of the paste upon a glass plate remains quite clear up to the congealing point and causes a slight burning sensation upon the tongue, the saturation of the paste with alkali may be considered complete (about 2500 lbs. of lye of 14° B. suffice for the complete saponification and saturation of 1000 lbs. of oil). The paste is now either boiled until entirely free from froth and then carefully separated, so that no froth is formed, with common salt or brine of 24° B., the result being a thick grain free from froth; or the paste is boiled not entirely free from froth but salted out when it spins threads, and after removing a portion of the spent lye the grain is boiled clear like tallow soap.

For mottled soap the grain slightly ground and colored is brought into the frame, thoroughly crutched by hand, and the frame covered. For smooth soap the grain is ground by crutching in hot water or, what is better, potash lye of 3° to 4° B. until it has acquired sufficient fluidity to allow all the impurities to pass into the spent lye.

Soap prepared from palm oil soon develops an agreeable odor of violets, and yields a thick, fat lather. One hundred pounds of palm oil give on an average 150 to 155 pounds of soap; this yield may be increased a few pounds by grinding.

Soap prepared from pure palm oil being somewhat hard and brittle, the oil is generally worked in connection with softer fats, such as bone fat, olein, lard, peanut oil, etc., or with 15 to 25 per cent. of rosin. Below, a few such soaps prepared

from palm oil with an addition of other fats or rosin will be described.

*Stettin palm-oil household soap.* Eight hundred pounds of palm oil and 200 pounds of bone fat and 500 pounds of lye of  $12^{\circ}$  B. are brought into the kettle and combined by crutching over a slow fire. As soon as the mass is thoroughly combined more lye of  $12^{\circ}$  to  $14^{\circ}$  B. is added, and the whole allowed to boil thoroughly, the addition of lye, with vigorous boiling; being continued until a pale, clear paste is formed and a sample of it brought upon the tongue shows a slight touch. For the saponification of the 1000 pounds of fat about 2500 to 2600 pounds of lye of  $12^{\circ}$  to  $14^{\circ}$  B. suffice. Now add to the saturated, clear soap-paste, with constant, vigorous stirring, in small portions, 150 pounds of pale comminuted rosin and 150 pounds of caustic soda lye of  $20^{\circ}$  B., and allow the whole to combine with gentle boiling. When, after a few hours, the paste is clear and shows a slight touch and a sample "wets" (shows moisture) and slides upon a glass plate, salt well out with brine of  $24^{\circ}$  B. and boil the resulting grain clear. Now cover the kettle, and after allowing it to stand for a few hours to give the spent lye time to settle, bring the grain into small, low frames so that it cools rapidly. The soap is cut into rectangular pieces and pressed on all four sides.

*Palm-oil rosined soap.* This soap has a fine waxy appearance, is readily soluble on account of the addition of rosin, yields a stiff lather and possesses good detergent properties. For its manufacture bring 425 lbs. of bleached and 75 lbs. of crude palm oil and 250 lbs. of tank lye or caustic soda lye of  $14^{\circ}$  B. into the kettle and combine them over a moderate fire. Then add more lye of  $14^{\circ}$  B. and increase the fire. After continued boiling and adding more lye, the mass gradually forms into paste, which becomes thicker and clearer the further its saturation with alkali progresses. The oil being completely saponified, which requires about 1250 pounds of lye of  $14^{\circ}$  B., and the thick, clear paste showing a slight touch, 150 pounds of comminuted, pale rosin and caustic-soda lye of  $20^{\circ}$  B. are

added in portions until the paste is thoroughly fitted, a very moderate fire being kept up during the operation. For the saponification of the rosin about 150 pounds of caustic soda lye of 20° B. will be required.

The sufficient fitting of the paste is recognized not only by a slight touch, but better and surer by a sample upon a glass plate, which, with thorough fitting, will slide upon the glass and show moisture.

The fitted paste is thoroughly salted out and the resulting grain, after pumping out a portion of the spent lye, boiled clear so that it lies thick and free from froth in the kettle and a sample shows solidity and pressure.

For the preparation of mottled soap the grain, after clear-boiling and settling of the spent lye, is brought into the frames and the latter are covered. The operation of mottling is then commenced in the same manner as described for tallow soap.

For smooth soap the clear-boiled grain has to be ground. For this purpose the greater portion of the spent lye is removed and the grain ground with hot water until the spent lye begins to thicken and the grain is completely dissolved and lies bright in the kettle. A sample should show sufficient solidity, but no touch. In grinding care must be had that the soap does not become too pasty or too soft. After settling and resting for some time, the ground soap is brought into the frames.

Some manufacturers prepare this soap by the following process: The palm oil is first completely saponified with lye of 14° B. The clear fitted paste is then salted out, and, after removing the spent lye, the rosin is added and saponified with caustic soda lye of 20° B. The soap, after salting out, is boiled until it is free from froth and clear, and a sample is solid with a good pressure. The soap is finally ground with some water. This process also yields a beautiful product.

*Palmitin soap.* The soap brought under this name into commerce by some manufacturers is prepared chiefly from palm-oil fatty acid in connection with a small quantity of

other fats. As fatty acids saponify very quickly, in preparing soap from them, a method of working somewhat different from that customary for neutral soaps may be adopted, and saponification can be best effected by stronger lyes containing more carbonic acid. In working with stock-lye the procedure is as follows:

The 2000 lbs. of stock-lye of 18° B. required for 1000 lbs. of stock consisting of 3 parts of palm-oil fatty acid and 1 part of saponified olein are brought into the kettle and heated to boiling. The above quantity of fatty acids is then added in portions, whilst constantly crutching, to the gently boiling lye and at once saponified. In adding the fatty acids the utmost care is necessary not to add a fresh portion before the preceding one has been completely absorbed by the lye. When a clear paste is formed, it is fitted to touch, allowed to boil for some time longer, and then salted out. The resulting grain is then boiled clear in the previously mentioned manner, and, after removing a portion of the spent lye, ground with water until a cohesive liquid mass is formed. The soap is now allowed to rest for twenty-four hours in the covered kettle, and then carefully pumped from the thinly-fluid paste beneath it into large frames and covered. When cut such soap presents a silvery radiated appearance; it also possesses an agreeable odor. The yield is about 160 lbs. of soap from 100 lbs. of fat-stock.

It is, however, more suitable to work according to the carbonate saponification process. The content in per cents. of the fatty acid has first to be determined and the consumption of soda ash calculated according to this, 21 per cent. of soda ash being allowed for every 100 per cent. of free fatty acid. Suppose the determination has shown a content of 90 per cent. free fatty acid, then there will be in a stock of 1000 lbs. 900 lbs. free fatty acid, for which  $900 \times 21 = 1890$  lbs. of soda ash are required. The soda is dissolved in sufficient water to make a solution of a density of 30° B. This solution is brought into the kettle and made boiling hot, when the fatty acid, prefer-

ably previously liquefied, is allowed to run in gradually so that one portion saponifies before another is added. When the last portion has been brought into the kettle and all is saponified, the mass is allowed to boil until the soap no longer rises and lies as a mass difficult to move in the kettle, puffing up with difficulty and a whistling noise; this indicates that all the carbonic acid of the soda ash has escaped. The 100 lbs. of neutral fat which have still to be saponified are treated with 50 lbs. of caustic lye of 40° B. The soap is then fitted to touch, salted out and, after removing the spent lye, treated with water so long as paste deposits.

*Olein.* Olein is obtained as a by-product in the manufacture of stearic acid from tallow. It has for many years been utilized as an excellent raw material in the manufacture of soap, the grained products prepared from it being especially liked in the cloth industry as well as for household purposes. As previously mentioned, two varieties of olein are found in commerce, viz., saponified olein and distilled olein. The former contains, as a rule, a few per cent. of stearic acid and gives quite solid soaps, the yield being about 150 lbs. of grained soap from 100 lbs. of stock. On the other hand, the soap prepared from distilled olein is often much softer and the yield frequently smaller. For this reason distilled olein is, as a rule, only used in connection with palm-kernel oil for smooth grained soap, while from a good grade of saponified olein, soap may be profitably produced without the addition of other fats.

The procedure of manufacturing pure olein soap is similar to that given for working olive oil and palm-oil fatty acid. The lye required should be high-graded and contain more carbonic acid, or consist entirely of soda solution. It is first brought into the kettle and the fat is then gradually added.

The simplest and most profitable method of manufacturing olein soap is by carbonate saponification, which has previously been described. When saponification and fitting have been effected, salt is added to the soap till a grain free from froth

is formed. The spent lye when thoroughly settled is pumped out or drawn off and replaced in the kettle by a few potfuls of lye of 7° B., the grain being then boiled for some time so as to become completely saturated with alkali. The soap is next ground with water so that the spent lye gets slightly pasty. The kettle is then tightly covered and the soap allowed to rest for 12 to 18 hours, when it is carefully ladled from the slight precipitate, brought into the frames, and the latter are well covered.

The soap thus prepared presents, when cut, a fine silvery-radiated appearance, and is also delicate and quite firm. If, however, the soap is intended to be cut into square cakes which are subsequently to be pressed or stamped, for which purpose they must present a perfectly smooth appearance, it is allowed to stand in the kettle for about 20 to 30 hours, and is then crutched in the frames until cold. If the firmness of olein grained soap is to be increased by the addition of some bleached palm oil, it can only be done when the strength of the strong lye at first present in the kettle has been absorbed by the olein; the palm oil, together with the requisite lye, is then added.

*Apollo soap.* The product known under this name is a ground olein soap. It is prepared as follows: The quantity of lye of 20° to 22° B. required for the saponification of the stock of olein to be worked is brought into the kettle, together with a certain quantity of tallow. The object of the addition of the latter is to prevent the mass from closing completely, since this would be a hindrance to the complete saponification of any olein which might still be wanting. The lye may be quite carbonated, such as is obtained of that strength in the tank with 45 to 50 per cent. of lime.

Lye too carbonated or pure soda solution would render saponification more difficult in consequence of too strong a development of carbonic acid and the heavy foaming-up resulting therefrom, and the whole operation would be much prolonged. For boiling 100 lbs. of olein 120 to 130 lbs. of lye

of 20° B. will generally suffice, and there will then be no loss of strength when the spent lye is removed. When the entire quantity of olein is saponified without the spent lye showing a biting sensation on the tongue, the kettle is tightly covered and allowed to stand, best over night, for the lye to settle. The next day the spent lye is pumped out or drawn off and, after adding some fresh lye, the soap is again allowed to boil up so that the grain becomes completely saturated, and the spent lye also shows a faint touch and is not a hindrance in grinding the soap. When the lye after boiling for some time shows still a faint touch it may be confidently assumed that the grain is completely saturated.

Clear-boiling is in this case useless and the operation of grinding can be at once commenced if the soap has not too much or only very weak spent lye. Steam, if available, is here very practical; it is at once introduced and the operation aided by warm water until quite a homogeneous and but slightly wetting soap-paste results, the soap globules form a firmly cohering mass, and the spent lye separates only with difficulty from a sample of the soap upon the spatula. When this is the case boiling may be considered as finished; the steam is then shut off or the fire withdrawn and the soap is allowed to rest for some time, larger boilings at least 36 hours, the kettle being well covered. Smaller boilings, up to from 4500 to 5000 lbs., are best brought into the frame the next day, so that they retain sufficient heat to recrystallize.

The soap should be carefully drawn off. If there is still a thin film of froth on the surface it is cautiously removed to prevent froth from getting into the frames. It is also necessary to see that none of the pasty spent lye reaches the frames, as this would cause wet, blind spots; it is much better that some grain should remain upon the lye than that any of the latter should get into the frames. After twenty-four hours the frames are well covered before they are allowed to cool.

The pasty spent lye is boiled with some bone fat, olein or other old fat and salted out and saved for a fresh operation.

It is a remarkable fact that a sufficiently firm soap resulting in a yield of about 150 per cent. can be prepared from saponified olein without the addition of harder fats, while a serviceable, salable product cannot be obtained by boiling distilled olein by itself. Some hard fat of a better quality is always required to give to the soap firmness and the proper capacity of crystallizing. However, as the distillation products vary very much, a definite proportion for the composition of a serviceable stock of fat cannot be given, it must be found by experiment. However, one-third, and sometimes even one-half, of the stock will have to be of a better quality of fat.

Pure olein grained soap, when fresh, is, to be sure, quite soft, of a pale brown color and a pleasant, sweetish odor, but it becomes firmer in drying and, with a loss of about 12 to 15 per cent. in drying, acquires sufficient hardness. By being stored for some time it fades and assumes a wax-yellow to pale yellow color. Grained soap, prepared as above described, is an excellent wool-milling soap, and also much liked for household use.

By the addition of one-third to one-half of bleached palm oil the hardness of such olein soap is considerably increased without changing its agreeable odor. The cost of production is, to be sure, considerably increased by such addition, but the value of the soap is also increased in the same proportion. The procedure of boiling such a soap is only changed in so far that the palm oil is previously saponified in the customary manner; when the grain is free and liquid the saponification of the olein is then finished, a somewhat stronger lye being at the utmost used. The procedure is similar with an addition of tallow, lard or bone fat.

*Grained soap from fuller's fat.* Partly on account of its large content of fatty acid this soap, which is prepared chiefly from fuller's fat, is much used in the woolen cloth industry and, when color and odor are not objectionable, also for household purposes.

Fuller's fat is recovered by decomposing the soap-waters

from cloth mills with sulphuric acid, a product of quite a light color being obtained after passing it through various operations. The value of fuller's fat varies very much, according to the quality of the soap used in the mills. The color of the fat is also of importance, as dark fat requires to be boiled upon several waters, and the appearance of the resulting soap is less desirable. Fuller's fat, being a pure fatty acid, is readily saponified in a similar manner as olein, and under certain conditions may yield a good grained soap. Generally, however, it is boiled in connection with other solid fats, the resulting soap having a better appearance and feel.

Soap from fuller's fat was formerly, as a rule, prepared by bringing about 1000 pounds of stock lye of 15° B. into the kettle, and, after heating to the boiling point, adding 50 to 60 pounds of common salt. The fire was then moderated and the fuller's fat (about 500 pounds) gradually introduced. In adding the fat great care had to be observed and water or weak lye always kept ready on hand, as on account of the escape of carbonic acid the mass might foam up and run over. Saponification was generally complete after crutting in all the fat. A small excess of lye is of advantage, as it promotes the decolorization of the fat.

The soap lying in the kettle as a small round grain was allowed to boil for some time with a moderate fire, when 50 pounds of comminuted rosin and about 40 pounds of caustic lye of 20° B. were added. Boiling was then continued until a round grain was again formed, which was fitted. After extinguishing the fire and allowing the sub-lye to settle, the soap was ladled into large barrels and the dirty spent lye allowed to run off. The soap, together with some other cheap fat and soap scrap, was then returned to the clean kettle, sufficiently evaporated by gentle boiling, and carefully and slightly fitted with caustic lye of 20° B. When a sample of the thick soap, after cooling, was dry and without sharpness the finished soap was brought into the frames.

The preparation of this soap may also be effected by car-

bonate saponification. The material in question being a fatty acid without any neutral fat, for every 100 lbs. of it 21 lbs. of soda ash are allowed. The latter is dissolved in water to form a solution of 30° B. and then treated as previously described under carbonate saponification. To every 100 lbs. of fuller's fat are at the same time added 10 lbs. of brine of 20° B., the washing and grinding of the resulting grain being thereby promoted. When all the carbonic acid has been expelled the soap is strongly fitted with caustic soda lye.

For the production of a paler, harder soap, for instance, for wool-milling purposes, the rosin is entirely omitted and, after removing the spent lye and thoroughly cleansing the kettle, about 250 lbs. of lye of 25° B. are added to the decolorized grain of fuller's fat. To the boiling grain are then added in portions about 100 lbs. of olein, and when this is thoroughly dissolved, 100 lbs. of palm oil or bone fat. The soap will soon appear thoroughly combined and fluid, and also boil high. It is then slightly fitted with lye of 25° B., next salted out, clear-boiled, and the thick grain free from froth is somewhat ground with weak lye.

*Soap from wool fat.*—A brown, sticky, and quite solid fat is obtained in washing raw sheep's wool with soap and decomposing the wash waters with acids etc., and brought into commerce under the name of wool-fat. It cannot be classed as an actual fat since, when boiled by itself, it does not yield a serviceable soap. At the present time its use in the manufacture of soap is limited; it is employed as an addition to stock for so-called economy soaps, chiefly for the purpose of preventing them from graining, and sometimes also for dark rosined soap.

The procedure of manufacturing a rosined grained soap with an addition of wool fat is in many cases as follows: 250 lbs. of bone fat and 50 lbs. of crude palm oil, together with about 150 lbs. of lye of 12° B., are brought into the kettle and combined by moderate boiling. When the mass is thoroughly combined the fire is increased and more lye of 12° to 15° B.,

is added in portions, until a clear paste free from froth is formed which is finished so as to show, when tasted, a faint touch. About 200 pounds of wool-fat and the required 200 pounds of lye of 22° B. are then added to the paste, and after thorough combination is established the whole is allowed to boil some time longer. Now slacken the fire and add gradually 50 pounds of comminuted rosin, together with about 40 pounds of caustic soda lye of 20° B., carefully fit the paste, after further boiling, with caustic soda lye of 25° B. until a sample shows moisture, and then salt out. The dark spent lye is now removed and a few bucketfuls (only sufficient to prevent scorching) of light-colored spent lye derived from grained soap are thrown into the kettle, and boiling is continued until a thick grain free from froth and without sharpness is formed. The soap is then brought into the frame, drawn through with a rod and well covered.

*Rosined grained soaps.*—On account of its excellent qualities, such as rapid saponification, ready solubility and better lathering of the soaps prepared with it, rosin has for many years been worked in large quantities in soap factories. It is especially employed to a great extent for grained soaps, and such rosined soaps, which according to the color and purity of the rosin used are of a lighter or darker appearance, are prepared by various methods, a few of which will here be described.

1. *Rosined grained soap from 100 parts fat and 30 parts rosin.*—An old method which was formerly much in use is as follows: Combine by moderate boiling 1000 pounds of tallow and 150 pounds of crude palm oil with 700 pounds of lye of 10° B. When combination is complete increase the fire and add gradually to the boiling mass lye of 12° to 14° B. until a clear paste is formed. When the paste is thoroughly saponified, continue boiling until it lies free from froth in the kettle, and a sample shows clear and solid upon a glass plate and causes a faint biting sensation when brought upon the tongue. At this stage salt out the paste and the result will be a thick grain

free from froth. Allow it to rest for some time in the covered kettle for the spent lye to settle.

In the meanwhile saponify in another kettle 350 pounds of rosin by gradually adding lye of 20° B. (about 650 pounds) until the complete saponification of the rosin is indicated by lye separating from a sample. As the separated lye absorbs all the impurities and the greater portion of the coloring substance of the rosin, the soap obtained is always of a light color.

The spent lye is now pumped off from the soap in the other kettle and the dirty lye from the rosin soap. Then add the rosin soap to the grained soap, and mix both by vigorous stirring or the introduction of steam. The soap is generally boiled upon a second water by liquefying the grain with lye of 6° to 7° B., then boiling for some time, and again concentrating it with lye of 2° to 3° B. so that a somewhat thin precipitate of paste is formed. After allowing the soap thus obtained to rest several hours it is framed; when cut it is of beautiful appearance and great solidity.

The manufacture of this soap is now frequently effected as follows: The stock of fat—tallow, crude palm oil, and eventually some palm-kernel oil—together with the rosin is brought into the kettle with about half the quantity of caustic soda lye of 25° B. required for saponification and combination effected with a weak fire. When this is the case more lye of 25° to 26° B. is gradually added and when the clear paste free from froth shows a slight touch, it is salted-out. If there is any scrap it is melted with a moderate fire and the soap is allowed to stand over night in the covered kettle for the spent lye to settle. The next morning the spent lye is removed and the soap is ground, being constantly crutched, with boiling water and a moderate fire, or eventually also with direct steam.

2. *Rosined grained soap from 100 parts fat and 40 parts rosin.*—A cheap rosined soap of fine quality is according to another method prepared as follows: 500 pounds of bone fat, 400 pounds of tallow, 100 pounds of crude palm oil, and 700

pounds of lye of  $12^{\circ}$  B. are brought into the kettle and intimately combined by crutching over a slow fire. The fire is then increased and lye of  $14^{\circ}$  B. is gradually added until a clear, homogeneous paste is formed. This will require about 1800 pounds of lye of  $14^{\circ}$  B. The addition of lye is best executed by allowing one portion to thoroughly combine with the mass before adding another. When a sample of the paste remains clear for some time on a glass plate and shows a slight touch, vigorous boiling is continued until the paste is quite free from froth. The paste is then salted out, about 8 to 10 per cent. of salt of the fat used being sufficient for the purpose. After thorough settling the spent lye is pumped out or drawn off. The caustic soda lye of  $20^{\circ}$  B. (about 400 pounds) required for the 400 pounds of rosin is then added and the soap boiled clear. The previously comminuted 400 pounds of rosin are then added in portions and saponified with a moderate fire. The soap is then boiled until it lies free from froth in the kettle and shows good pressure. It is then thoroughly fitted and separated, and finally somewhat ground with water. The saponification of the rosin with strong lye is of advantage in itself because by the great content of water which, with the use of weak lye, is naturally brought into the soap, the formation of froth is readily promoted.

3. *Rosined soap from 100 parts fat and 50 parts rosin.*—Nine hundred pounds of tallow, 100 pounds of crude palm oil and 500 pounds of pale rosin are brought into the kettle and combined with 600 pounds of caustic soda lye of  $15^{\circ}$  B. with the use of a moderate fire. To the thoroughly combined mass add, while boiling vigorously, more lye of  $15^{\circ}$  B. until a quite clear paste has been formed. Then add caustic soda lye of  $25^{\circ}$  B., and boil until the paste lies free from froth in the kettle, and a sample pressed in the palm of the hand leaves moisture behind, this being a better and surer test than that by the tongue of the complete saturation of the rosined soap paste. The paste is then salted out, the soap boiled down thick and clear and the resulting thick grain ground with

some hot water. The soap is allowed to stand for about 12 hours in the covered kettle, is then brought into the frame and eventually crutched cold in it.

4. *Rosined soap from 100 parts fat and 80 parts rosin.*—Boil, the same as the preceding soap, 1000 lbs. tallow and 800 pounds rosin with caustic soda lye of 20° to 24° B. to a clear paste, which is fitted so that when tasted, it shows a faint biting sensation on the tongue. The paste is then salted-out until when taking samples the spent lye runs off clear from the spatula. The soap is then boiled for some time longer until there is a clear grain. It is now allowed to stand in the well-covered kettle for about 24 hours to settle. The spent lye is then removed and the soap ground with hot water until it is sufficiently liquefied. To make the soap firmer and to guard against its washing away too quickly, about 35 per cent. of silicate of soda (water glass) is crutched in. The soap if properly constituted should show a net-like appearance when crutting in the silicate of soda, and then does not deposit a sediment.

5. *Rosined soap from 100 parts fat and 100 parts rosin.*—Nine hundred pounds of tallow, 100 lbs. of crude palm oil and about 600 lbs. of lye of 12° B. are brought into the kettle and intimately combined with the use of a moderate fire. More lye of 13° B. (about 200 to 300 lbs.) is then added to the combined mass, while vigorously boiling, till a good clear soap-paste is formed. After withdrawing by continued boiling superfluous water from the paste, and the latter lies free from froth in the kettle and shows, when tasted, a slight touch, it is salted out. The spent lye is then removed, and to the grain in the kettle are added about 900 lbs. of caustic soda lye of 25° B. and gradually the 1000 lbs. of rosin previously comminuted. The whole is then boiled for some time so that the rosin becomes thoroughly saponified. By fitting with lye of 25° B., so that when tasted the soap shows quite a strong touch, a quite firm product is obtained. The spent lye is then again removed, the soap thoroughly ground with hot water,

again fitted and then left warm in the covered kettle to rest for 24 to 36 hours. The soap is then brought into the frame, and having become somewhat soft by grinding, solution of soda crystals of 35° B. (about 5 to 6 lbs. to 100 lbs.) is crutched in, whereby the phlegm is drawn and the soap becomes firmer. The soap is then crutched cold. Sometimes 1 to 2 lbs. of silicate of soda are added to the soda solution to prevent effervescence of the soda, it being also of advantage to the appearance of the soap.

*Transparent rosined soap.*—In some factories this soap is prepared as follows: Five hundred and twenty pounds tallow, 80 pounds crude palm oil and 400 pounds pale rosin are melted together and some scrap dissolved in it. About 1000 pounds of stock soda lye of 20° B. are then crutched in and, when a good pasty combination has been effected, more stock lye of 28° B. is added till the glass-test shows the settling of paste. The soap must not be made too hot, as otherwise it becomes frothy, and it should also be not too much grained, as otherwise it does not become transparent. When the glass-samples lie as thick as the back of a knife and, when cold, small gray-black dots are noticed in them, graining must cease. The soap is constantly crutched; when finished the kettle is well covered, the soap allowed to rest for some time, and then framed.

*Black rosined soap* is prepared as follows: 270 pounds palm-kernel oil, 30 pounds tallow and 60 pounds rosin are boiled with about 360 pounds of caustic soda lye of 25° B. to a clear paste. The paste, after adding 15 to 18 pounds of "goudron,"\* is well fitted, boiled free from froth, and then grained with strong lye or salt. When thoroughly settled the strong lye is removed and the grain ground with some hot water whilst being constantly crutched. The soap is then brought into the frame and crutched cold.

Such soap may also be prepared as a grained soap from ful-

\* Distillation-residue in the manufacture of stearin.

ler's fat, bone fat, dark tallow, palm-kernel oil, black cotton oil foots and dark rosin with caustic soda lye of 25° B.; 5 per cent. goudron may eventually be also used.

Various methods of boiling rosined soaps having been given in the foregoing, it remains to say a few words regarding the manufacture of these soaps. First, it is always of advantage to use pure fat and rosin. For this purpose it is best to dissolve the fat upon water and dissolve the rosin in the hot fat. After such an operation it will generally be found that the water is not only much colored, but that a thick sediment has formed upon the bottom of the kettle. Secondly, it is necessary in order to prevent the formation of froth to saponify the rosin with strong caustic soda lyes, and besides care must be had to saturate the rosin by a successive addition of lye. Thirdly, the rosined soaps must be only slightly fitted, since soaps with an excess of soda decompose. For a similar reason the use of caustic soda lye for rosined soaps is recommended, as this will frequently prevent sweating, for only soaps containing sodium chloride or much sodium carbonate, which attract moisture from the air, sweat.

*Turpentine soap.*—The cheapest and most convenient way to prepare this soap is to subsequently crutch a few pounds of turpentine into a rosined soap, which may be boiled clear upon spent lye or upon paste.

Ordinary rosined soaps, to be sure, are frequently sold as turpentine soaps, but if the odor is to be plainly perceptible and the soap is to be used for the removal of stains, it is best to prepare it by itself, it costing no more, but rather less, than other soap. An ordinary rosined soap is brought into a small frame of about 1000 lbs. capacity and 27 lbs. of soda crystals dissolved in a small quantity of water are crutched in; some silicate of soda may be used in place of soda. When all is thoroughly combined, which is recognized by the doughy appearance of the soap, 5 to 6 lbs. of turpentine are stirred in. The frame is then allowed to stand covered for a few hours.

*Russian saddle soap.*—Genuine Russian saddle soap is sold

in boxes. It is a rosined tallow soap which may be prepared by carefully melting Russian tallow and dissolving in it 50 per cent. of rosin. This mixture is saponified with one-half potash lye and one-half soda lye (stock lye of 24° B.). The resulting paste is allowed to settle and the grain ground with some warm water. The clear soft soap is brought into boxes or barrels.

*A good saddle soap in bars* is prepared as follows: 10 parts tallow, 5 parts crude palm oil and  $3\frac{1}{2}$  parts pale rosin are boiled to grain. The spent lye is then removed and about 7 parts of caustic soda lye of 27° B. are added, and next successively about 7 parts palm-kernel oil to cut the strength of the lye. The soap is then boiled thick, fitted so that when tasted it shows a faint biting sensation on the tongue, and by the addition of brine of 20° B. is brought so far as to be sufficiently fluid and thoroughly moisten the finger sample.

#### *b. Settled Grained Soaps.*

The preparation of smooth grained soaps, which are found so much in commerce, is effected by two different methods, according to the fats used. The object of both methods is to produce as pure a soap as possible by separating all the impurities with the superfluous content of water and alkali.

One method is to grind by means of water grained soaps boiled upon spent lye until a paste is formed; this is used for all soaps from animal fats, olive-oil, palm-oil and olein. The other method is based upon the formation of a precipitate of paste, which is produced by adding either lye or salt in excess, though in both cases not sufficient to effect complete separation of the soap. This method is only applicable when working cocoanut-oil or palm-kernel oil in connection with other fats, and can be executed either in a direct or indirect way.

Since, generally speaking, but few settled soaps are at present boiled without the use of palm-kernel oil, the latter frequently forming even the preponderating portion of the soap-

stock, the last mentioned method of working is commonly adopted. The entire soap-stock together with any additional fats or olein is saponified with caustic soda lye of 29 to 36° B., according to whether working with steam or an open fire, and fitted to a moderate touch. The soap is then moderately grained with brine of 24° B. up to the point of the separation of paste. With the method of boiling with pure caustic lyes, now in general use, it is not advisable to bring about the separation of paste by means of an excess of caustic lye, as in the presence of too much water mottled soaps are readily obtained, and soaps that are short and brittle in the outside cuts; besides they do not settle well and clean, and moreover possess a larger excess of free alkali.

Hence, in view of the present use of very pure raw materials and thoroughly caustic lyes, it is best to work with lyes as strong as possible, fit to a moderate touch, and slightly grain the soap with strong brine. The production of light-colored soaps free from froth and stains with as little paste as possible depends on the more or less careful observance of these three points, provided of course that the raw materials used and the rosin eventually employed are pure and of a pale color. If, on the other hand, darker fats are to be worked together with the soap-stock it is, of course, more correct to first boil them together with the rosin with weaker caustic lye and bring them as a salted-out grain into the soap. By this preparatory boiling such fats are partly purified and decolorized, and the pale color of the soap is thus less impaired. Any paste-grain on hand may also be melted in this preparatory boiling, it being also improved by such remelting.

In view of the frequent fluctuations in the fat and oil market it is not an easy task to always obtain a uniformly fine product. It is therefore all the more necessary for the soap-boiler to be thoroughly acquainted with the qualities of all the fats and oils to be worked for the time being, and the best methods for their saponification, so as to treat them conformable to their nature and to gain from them the best soap possible.

All settled soaps must be free from dirt and be glossy. A product of a dead or dirty appearance cannot be considered of good quality even if its content of fatty acid is greater than that of a bright silvery-radiated soap. Hence for the soap stock, fats free from dirt and of as pale a color as possible should be selected and, if necessary, purified by boiling with salt water. If this should not prove sufficient they may be further improved by the above-mentioned preparatory boiling to grain. Direct boiling is, however, the method most generally used, it possessing the advantage of being quicker, though whether it is cheaper, as it would appear to be, is doubtful. Generally speaking, too much value is, without justification, set upon the finishing of a boiling of a soap as quickly as possible. In four to five hours from the time the soap-stock is introduced, and frequently in a still shorter time, it must be finished, the result being that in about fourteen days the greater portion of the soap, and often all of it, has to be returned to the kettle on account of its being defective. By boiling with pure material and strong lyes, the whole operation can to be sure be forced very quickly, but slower, quieter boiling is by all means to be recommended, since saponification thereby progresses more intimately and more thoroughly, and the soaps turn out better and firmer.

Boiling is a chemical process which is the more completely carried through the more time is allowed for its full development. Soaps finished in the shortest time are seldom of the best quality, and the occurrence of so many soaps which in a comparatively short time show, especially on the surface, uncombined fatty acids has without doubt to be attributed to their having been too quickly finished.

Although, as previously mentioned, direct boiling is at present almost generally preferred and is decidedly warranted with soap-stocks consisting entirely or almost so of cocoanut oil or palm-kernel oil, it may at times happen that other fats and oils, such as bone-fat, peanut oil, cotton-seed oil, etc., are cheaper. Larger quantities of such material will then of

course be used as additions to the stock, and in that case it is by all means more correct to subject such fats to a preparatory boiling, as the resulting soaps will be not only of a lighter color but also firmer.

The quantities of such additional fats which may be used vary much according to their kind, since the greater or lesser firmness of the soap depends thereon, and on the other hand, the property of lathering readily, which is characteristic of all cocoanut oil and palm-kernel oil soaps, is decreased by too large an addition of other fats. Thus, for instance, very firm soaps of good appearance can be prepared from a good quality of beef-tallow with additions of only 10 to 12 per cent. palm-kernel oil; but notwithstanding their economical consumption, such soaps are not liked by consumers because they give too little lather. Although tallow is almost always more expensive than palm-kernel oil it may under certain conditions be desirable to work it to a greater extent. In such a case it is advisable to maintain the soap stocks so that they always contain 50 per cent. palm-kernel oil; this applies also to the use of bone fat, horse fat, or similar fats as additions. If the soaps may or are to contain rosin, the addition of palm-kernel oil may be reduced to 25 per cent. and soaps lathering quite freely may also be prepared from tallow and 15 to 20 per cent. rosin, but such combinations do not yield soaps lathering as freely as demanded by many consumers. Hence it is well not to boil soap-stock with less than 25 per cent. palm-kernel oil, leaving out of consideration the additions of rosin. When larger quantities of tallow or tallowy fats are worked, it is advisable to first boil them to grain as better saponification is thus attained and what has to be especially taken into account with softer fats, the resulting soaps are firmer. If on account of their lower price cotton seed oil or peanut oil is to be used not more than 33 per cent. of them should be added with due regard for the firmness of the soaps, and if the latter are to be rosined it is well to limit the addition of such oils to 25 per cent. This also applies to additions of linseed oil in times when it is especially cheap.

Cotton-seed oil and linseed oil are drying oils, especially the latter. Soaps with such additions give lower yields and are subject to the danger of becoming yellow- or brown-spotted after being stored for a shorter or longer time. The opinion is frequently expressed that the soap can be protected from such spotting by being strongly fitted. Such, however, is not the case, for it has been proved by practical experience in working such oils that moderately to slightly fitted soaps keep longest before the formation of spots appears, while stronger fitting seems to favor their earlier formation. Of the two oils named linseed oil is the most sensitive in this respect and gives also the lowest yields; cotton-seed oil is now of better quality than formerly and is less sensitive.

The better grades of cotton-seed oil, preferably that from hulled seed, can to a limited extent be used as an addition in the preparation of settled white wax grained soaps. Thus, for instance, soap-stock of 2 parts palm-kernel oil and 1 part each of a good quality of white tallow and of American cotton-seed oil from hulled seed would yield a fine white grained soap. The tallow together with the cotton-seed oil should be boiled to grain with caustic lye of 15° to 18° B., fitted so as to show a scarcely perceptible biting sensation on the tongue, and then salted out. After removing the spent lye the caustic lye of 24° to 25° B. required for pasting the palm-kernel oil should be added and boiled, according to conditions, over an open fire or by steam. In boiling with steam some water always gets into the kettle and it is advisable to use lyes of somewhat greater strength—about 27° to 28° B. In calculating the lye it is well to always start with the consumption of caustic soda; the lyes can then be made according as they are to be used. It may be estimated that 100 pounds of any kind of fat require for saponification 17 pounds of caustic soda, this quantity giving a small excess of alkalinity even when working cocoanut oil, while this excess is still larger with tallow, peanut oil, cotton-seed oil, and other fats. When the grain together with the lye has been brought to the boil, the palm-kernel oil

is gradually added so that one portion is completely saponified before adding another, the sudden saponification of a large quantity of palm-kernel oil and consequent energetic rising-up or foaming being thus avoided. When all the oil is in the kettle and combination complete, there should be a quietly boiling soap in the kettle which should show but a moderate touch, but nevertheless have good pressure and sufficient firmness. By carefully separating at this stage the soap with brine of 24° B., so that the separation of the paste from grain can be noticed, a pure, thoroughly settled soap, free from stains, from the top to the paste bottom, will be obtained.

Frothy, dirt-stained soaps are evidence of too strong fitting, and this shows the necessity of a more energetic separation with brine. Perfectly caustic-boiled soaps require no excess of alkali, at least none that can be noticed on the tongue. The more accurately this is managed, the less brine will have to be used for separating the soap, and the more free from froth and dirt the latter will be obtained.

Soaps fitted strongly and to touch lack, when purely caustic-boiled, fluidity and mobility. They are viscid, pasty and thickly fluid, and for this reason do not allow the dirt and coloring matter to fall to the bottom at all or only insufficiently so. They are especially dirt-stained and spotted when too much brine has been used for separation, which, however, becomes necessary when fitting with weak lyes to a strong touch. With the pure caustic lyes of the present time the conditions for obtaining fine settled soaps are not to work with too weak lyes, and to restrict the quantity of lye for fitting to what is absolutely necessary, the latter provision applying also to the use of common salt. It should always be borne in mind that while for the formation of well-combined, serviceable and durable soaps all fats, to be sure, require complete saturation with alkali, a larger excess of uncombined alkali is not only superfluous but absolutely injurious, because it only remains mechanically mixed with the soap, and for that reason causes disturbances. In boiling with pure caustic soda any quantity

which shows a perceptible biting sensation on the tongue must be designated as a larger excess.

After these general remarks the boiling of soap from the above-mentioned materials will be described by giving the separate operations in detail.

*Wax soap.*—Suppose 5000 kilogrammes \* of soap-stock consisting of 1250 kilos beef tallow, 1250 kilos cotton seed oil from hulled seed and 2500 kilos palm-kernel oil are to be worked into smooth white wax soap. As 100 kilos of oil and fat require for saponification 17 kilos of caustic soda,  $50 \times 17 = 850$  kilos of caustic soda are first dissolved. As a drum generally contains 250 to 300 kilos of caustic soda, three drums w<sup>ould</sup> be sufficient. It is, however, advisable to dissolve, as a precaution, the contents of four drums so as to have a supply of caustic lye for any contingency which may arise. Next determine the weight of each drum and deduct 10 kilos tare from each drum. Suppose the net weight is 1125 kilos, which multiplied by 2 gives 2250 kilos. The bottoms of the drums are removed by means of a chisel and also the covers of the filling apertures in the heads. The drums are then hoisted by means of a tackle into an empty lye-tank. Since 1 kilo of caustic soda when dissolved in water gives 3 kilos of lye of 38° B., 2250 kilos of cold water are brought into the tank, and the whole is allowed to stand till the next morning, when the soda will be found dissolved. The empty drums are then removed and the lye is thoroughly crutched. The result will be 3375 kilos caustic lye of 38° B. As for boiling 250 kilos of tallow and cotton-seed oil 125 kilos lye of 38° B. are required; this quantity is brought into another tank or into a moveable iron kettle which is placed alongside the soap kettle. Enough water is then added to this lye to reduce it to 15° or 16° B. About 50 kilos of this lye are then brought into the soap kettle and, when working with an open fire, an equal quantity of water is added. The

\* 1 kilogramme = 2.2 lbs.

tallow and cotton-seed oil are then brought into the kettle and the fire is started, or steam admitted. The fats while melting combine simultaneously with the lye, and in a short time 50 kilos more of lye can be added. Firing or steaming is now continued for some time longer until a peculiar thumping of the mass of fat calls attention to the fact that more lye is needed, when 50 kilos more of it are added and so on until a well-combined pasty soap boils in the kettle. When there are still 100 to 150 kilos of lye available, the sample of soap taken by means of a paddle is more closely examined and, with some practice, it will readily be seen whether the soap is almost saturated with alkali, or whether it still requires a larger quantity of lye. However, great care has to be exercised, as a soap showing a perceptible biting sensation on the tongue is not wanted. Should this nevertheless happen, it is neutralized with about 100 kilos of tallow kept in reserve for this purpose from the soap-stock. In the meanwhile dry salt has been got ready and is gradually scattered over the boiling soap in the kettle. In observing the effect of the salt upon the soap, it will be noticed that the latter commences to tear and that spent lye separates. The latter should remain perfectly clear, must not be turbid or, in cooling, become jellied. If the latter were the case it would indicate that uncombined alkali is still in the soap and 50 to 100 kilos of tallow or cotton-seed oil would have to be brought into the kettle and boiled with the rest. The lye must be absolutely clear and not have a pasty appearance. When spent lyes become pasty it can always be traced to a content of caustic alkali or of alkaline carbonate, and it can only be remedied by boiling with fat or fatty acids, but never by strong salting out.

When finally the spent lye is clear the soap is allowed to stand quietly for about two hours without fire or steam, the spent lye during this time settling to the bottom. If there is another soap-kettle it is now made ready; it should, of course, be clean. The lye necessary for the saponification of the

palm-kernel oil is now brought into the kettle, 1250 kilos of 38° B. being required for this purpose, which are reduced with water to a density of 25° to 27° B. In the meanwhile the grain in the other kettle has become quite free from lye. It is carefully ladled out and brought into the other kettle, the fire under the latter being at the same time started. A quantity of palm-kernel oil is now from time to time added till everything is saponified and the soap itself boils quietly without perceptible rising in the kettle. When the soap thus boils quietly and it may be assumed that all the palm-kernel oil has been properly saponified, the fitting is tested. An experienced soap-boiler can immediately judge by the feel and finger-pressure of a cooled sample whether the soap is sufficiently fitted or whether it is still too weak; but the beginner will have to be also assisted by the tongue to determine the state of fitting. When conversant with this test he may commence such corrections as may be required as to whether lye has to be added or whether too large an excess of alkali has to be cut with oil or fat. If he notices a perceptible biting sensation on the tongue, 100 to 150 kilos of oil have to be immediately added and thoroughly boiled for at least half an hour. When this has been done another test is made, and testing repeated till no biting sensation is noticed on the tongue.

Phenolphthalein solution is now used for further testing. The solution itself is colorless, but turns red when brought in contact with alkalies. If a drop of it is allowed to fall upon soap to be tested, the latter turns blood-red if an excess of alkali is present, but remains colorless when fitting is still wanting. Hence, with the assistance of this reagent it can be readily and accurately determined whether all the fat has been saturated with alkali or whether an excess of it is already present. An excess of alkali traceable by this reagent is sufficient proof of sufficient fitting. However, this test is not reliable where boiling is effected with stock lye, as alkaline carbonates also react with it; neither is it available for soft soaps.

The soap is now allowed to boil for about one hour longer in order to be sure that the excess of lye present has been again boiled away, and the above mentioned test is for this reason several times repeated. If the result remains the same, the separation of the soap with brine of 20° B. is commenced, but very little of it will have to be used, because the soap having but a slight excess of alkali separates readily and deposits the paste. About 250 to 300 kilos of brine of 20° B. will suffice for the purpose.

If the soap after standing quietly for one hour is ladled out and covered in the frame, a mattress being wrapped round the latter, a product pure and smooth to the paste-bottom is obtained. If however, it is allowed to stand for 24 hours in the well-covered kettle the pure clear soap can be ladled from the paste and when this is carefully done, there will, after cooling, be found a frame of soap which when cut will be fit for use from top to bottom. The paste remaining in the kettle is boiled out with about 200 to 250 kilos of fat or tallow and, without the further use of salt, clear spent lye very pure and containing glycerin is obtained. Generally speaking the content of glycerin in the spent lyes of soaps thus treated amounts to between 7 and 8 per cent. according to the fats used.

The boiling of *settled rosined soaps* is effected in the same manner as above described, the only difference being that the rosin is also preparatively boiled in the grain, it being by this means to some extent discolored, and the dirt and coloring matter washed out. If no fats or oils are to be preparatively boiled, the entire soap-stock may be directly worked, using, however, lyes of 30° B. Fitting, graining the soap, etc. are effected as above described.

The demands made on a white grained soap as regards a more or less white color vary according to localities. While some consumers insist on an almost snow-white color, others are satisfied with a somewhat more yellowish shade. The whitest soaps are without doubt obtained from mutton tallow and cocoanut oil, but this is of coarse possible only when

adequate prices are paid for the soap. However, these raw materials yield also soaps which readily tear; this can, however, be prevented by including in the soap-stock some white cottonseed oil or peanut oil, 10 to 15 per cent. of either oil being sufficient for the purpose. Such a soap-stock might consist of: 15 parts oil (either peanut or cottonseed oil) 45 parts mutton tallow and 40 parts cocoanut oil.

Beautiful white soaps can also be produced from soap-stock consisting of equal parts of beef tallow and palm-kernel oil; the tallow must, of course, be fresh and white. When a good quality of light-colored horse fat, lard or American white bone fat is available, such fat may be substituted for a portion of the tallow. However, tallow, together with cocoanut and palm-kernel oils, is after all the best raw material for obtaining the finest qualities of white soap.

A particularly fine and white grained soap is obtained by working tallow and palm-kernel oil according to the following method: The tallow is killed and thoroughly boiled with caustic soda lye of 12° to 15° B., very slightly fitted and salted out; the spent lye is then removed. To the grain in the kettle is then added the quantity of caustic soda lye of 25° B. required for boiling the palm-kernel oil. The grain is boiled with this lye and when the palm-kernel oil has been gradually added it is fitted to a moderate touch, again salted out, and the spent lye removed.

Sufficient water is added to the salted-out grain to change it to a paste and boil up like a thin, strongly-ground soap. Soap thus treated possesses the power of depositing any light particles of dirt still inherent to it in the paste or to throw it into the light cover of froth. The soap is allowed to stand over night in the well-covered kettle. The next morning the light skin of froth on top is removed and the transparent grain lying beneath it carefully lifted from the paste. When cutting the soap it will from top to bottom be found pure and free from defects. Not every soap boiler will of course spend so much labor on one kind of soap, but the trouble is amply re-

paid by the larger quantity of salable first-class soap obtained as compared with a product prepared with less care and expense of time, which gives more scrap and paste bottom, and besides turns out greyer and less white.

*Oranienburg grained soap.*—This soap, frequently also called *olein soap*, is a smooth rosined grained soap. It is generally demanded of a very pale, waxy, yellowish color. The palest brands of French or American rosin should be used, and fats which yield clear white soaps, such as white palm-kernel oil, tallow, cotton-seed oil, white olein, lard or lardaceous fats, light-colored bone fat, etc. It is advisable to use as soap-stock at least two-thirds palm-kernel oil and one-third of the above-mentioned fats. Suitable stocks are, for instance : Palm-kernel oil, 1200 pounds ; tallow or light-colored bone fat, 400 pounds ; cottonseed oil, linseed oil or pale olein, 200 pounds ; pale rosin, 360 to 450 pounds—hence 20 to 25 per cent. of the fat ; or : Palm-kernel oil, 400 pounds ; fats (horse fat or light-colored bone fat), 200 pounds ; cottonseed or linseed oil, 200 pounds ; pale rosin, 120 to 160 pounds.

It may happen that lots of cocoanut oil may be bought cheaper than palm-kernel oil. Although in such a case the oil will, as a rule, be old and contain much free fatty acid, cocoanut oil is very suitable for settled soaps, and at the same price deserves the preference over palm-kernel oil, as a somewhat larger quantity of soft fat or oil can be worked with it without the soap turning out softer on that account. An excellent product, pale as well as firm, is obtained from stock consisting of : Cocoanut oil 400 lbs., linseed or cotton-seed oil 200 lbs., pale rosin 90 lbs.

Linseed oil and cotton-seed oil can only be worked to advantage in connection with other fats which by themselves give firmer soaps, such as cocoanut oil, palm-kernel oil, tallow, etc. Besides, their use can only be recommended when they are very low in price, as the yield of soap is far less than from other fats, which is the case to a still greater extent as regards castor oil, which, moreover, gives very soft soaps, so that its use is absolutely unprofitable.

All scrap, as well as the product resulting from boiling the precipitated paste of previous boilings of the same kind of soap, are brought into the kettle and boiled with lye of at least  $29^{\circ}$  to  $30^{\circ}$  B. until all froth has disappeared, the quantity of lye being about one-half of that of the stock. Combination is then carefully induced with the palm-kernel oil stock. The larger part of the rosin previously comminuted is held in readiness to promote combination, it generally taking place when about one-half of the rosin has been added. The rosin also prevents the mass from rising too violently when combination takes place, lye being also held in readiness to prevent boiling over. Caution is always necessary in effecting combination with such strong lyes.

When combination is complete and the mass boils quietly and uniformly, the quantity of lye still wanting is gradually added together with the rest of the stock, *i. e.*, the fluid andlardaceous fats, until the soap boils thick and curly, but shows a perceptible touch. With a moderate fire the rosin still wanting is then allowed to dissolve and the mass again fitted to a good caustic taste. With an average lye of  $30^{\circ}$  B. the soap can now scarcely boil excessively thick, because the more rosin is worked the thinner the soaps turn out and the stronger the lye required towards the end of boiling must be without the necessity of additional salting or separating with some rosin. A slight excess of lye perceptible upon the tongue or by turbid dull streaks upon the paddle suffices for the separation of a paste precipitate. If, however, the soap should boil somewhat too thick and heavy, a few bucketfuls of water, weak lye, or brine suffice to render it more loose and thinner. Light, loose boiling is an indication that the soap is finished. The fire is then removed, the kettle covered for a few hours, and the soap finally framed while hot. The frames remain covered for two days for the paste to settle.

When boiling is effected as above described there remains in the kettle but a very small quantity of paste which, when boiled with some olein or bone fat and then salted out, gives

very little spent lye. The more viscid and firmer such paste precipitate is—which is attained by avoiding salt as much as possible and not fitting excessively sharp—the more can good settling in the frames be counted on. Such paste precipitate can, when cold, be drawn like leather from the grained soap in it, the latter being pure and salable to close above the paste.

Soap boiled according to the above described process will never show froth, as the formation of the latter is from the start prevented by the use of lye of sufficient strength. Hence it is far more expedient to be obliged to add some water or weak lye towards the end of the boiling than to be forced to evaporate perhaps for hours, which moreover does not always prevent the formation of froth. This method offers the further advantage of not having to drag large quantities of paste-precipitate from one boiling to the other as is so frequently the case even in factories manufacturing every week large quantities of these soaps. Besides large boilings can be faultlessly finished in a comparatively short time over an open fire without the use of steam.

All soaps boiled from stock the greater portion of which consists of palm-kernel oil, have a tough, but fine and lustrous formation of grain-fiber and can only be cut by a well-constructed slabber, such a machine being indispensable when working on a large scale.

The yield, inclusive of the grained soap recovered from the precipitate and counting the rosin as fat, is about 150 per cent., which might be somewhat increased by the use of larger proportions of tallow, palm oil, or good qualities of lardaceous fats in the stock, while it might turn out somewhat less with the use of a greater proportion of fluid vegetable fats such as linseed or cotton-seed oil.

Besides the use of a larger proportion of linseed and cotton-seed oils in the stock for Oranienburg soap is not without influence upon the boiling and turning out of the soap. If, for instance, only palm-kernel oil perhaps 75 per cent. and cotton-seed oil 25 per cent., or the same quantity of linseed

oil are used for stock, the soap, with the same strength of lye, boils much thinner and has to be evaporated, or somewhat stronger lye has to be used in finishing. Nevertheless there remains a larger paste-precipitate, the soap turns out somewhat softer and remains so even when well dried. Linseed oil is especially sensitive to brine or an excess of lye, a lye-bottom instead of a paste-bottom being in consequence frequently formed in the frame, and wet spots in the soap.

*Rosined grained soaps.*—This term is frequently applied to soaps which are prepared in the same manner as Oranienburg soap, but contain more rosin—up to 30 per cent.—and are colored red with crude palm oil. Darker rosin may also be used for them. As a rule, or at least in many cases, these soaps are considered as objects which allow of the utilization of darker fats—perhaps foots of better quality—as well as the paste-bottoms of Oranienburg soap and other soap of a darker color, as much is covered by the color of the palm oil. It is however advisable not to go too far with the use of such waste and scrap, as the consumer demands the color to be as pale and pure red as possible. Hence these soaps are found in commerce of the palest yellow to the deepest red-brown color, and corresponding prices are paid for them. Generally speaking, it is easier to obtain better prices for Oranienburg as well as rosined grained soaps of a light color, than to dispose of the darker qualities of both kinds at a lower price. The boiling of rosin grained soaps is entirely analogous to that of Oranienburg soap. However, on account of their greater content of rosin, more of such fats which yield firmer soaps, such as tallow of inferior quality, palm-kernel oil, bone fats of firmer consistency, are used for their preparation. The coloration of the soap is effected with 5 to 15 per cent. of palm oil of the stock used. It is not advisable to use a larger quantity, as otherwise in using the soap, the wash might become colored.

*Filling grained soaps.*—As regards grained soaps, a larger yield is, as is well known, obtained by grinding, the soaps

absorbing water during this operation. The content of water thus incorporated should, however, not exceed a certain limit, as otherwise the soap becomes too pasty and soft.

The yield of grained soaps and rosined grained soaps, especially those boiled clear upon spent lye, is frequently considerably increased by crutting in various substitutes or, as it is technically called, by *filling*.

For such an artificial increase in yield, materials must, of course, be selected which do not injure the appearance of the soap to a great extent and do not dry too quickly. As especially suitable filling materials, silicate of soda, soda solution, and talc are recommended; they are generally used by themselves or in connection with each other. Silicate of soda used by itself for filling gives to soap in a fresh state a good appearance and protects it from drying out too rapidly; however, when the soap is thoroughly dried out it is unsightly and becomes stone-hard. If silicate of soda is to be used, it is best to employ a solution of it of 24° to 28° B., prepared by diluting silicate of soda of 33° B. with soda or potash lye of 2° B. On the other hand, grained soap filled with soda solution alone dries out very much, while talc, well distributed, protects it from drying out too strongly and becoming hard, but imparts to it a duller appearance. When used together these materials act supplementary to each other, and an excellent filling composition suitable for most grained soaps is prepared by stirring 200 pounds of talc into 300 pounds of boiling water, then adding 60 pounds of crystal soda and gradually crutting in 280 pounds of silicate of soda.

For the preparation of grained soaps which are to be filled it is best to use fats rich in stearin, such as tallow, palm oil, fatty acid, etc. The fats are, as previously described, first formed into paste with suitable lyes, and then evaporated to a stiff grain, free from froth, which is thoroughly ground with water. Previous thorough saponification of the fats used is absolutely necessary, as the soap, if it shows any defect, can be filled only slightly or not at all. When the grain is suffi-

ciently ground and dissolved and shows good pressure while the spent lye remains clear, the kettle is covered and allowed to stand for some time for settling. When the soap has cooled down to between 167° and 170° F. all the spent lye is removed, and from 30 to 40 lbs. of the above filling material for every 100 lbs. of fat used are crutched into the soap. As the soap to be filled does not always turn out alike, and therefore will hold more or less filling material, it is advisable, so as not to endanger an entire boiling, first to test the holding capacity with small quantities of soap.

Good results in filling can only be obtained by great care and experience. Many soaps fill more readily and better if the filling material is used warm, while others better absorb cold filling. The warm filling more frequently tears the soap asunder, while the cold filling is well absorbed. After the filling has been crutched in, the soap should be thick and bright. The operation of filling itself may be executed in the kettle after the spent lye has been removed, or in a suitable vessel.

In the same manner as grained soaps from tallow and palm oil, rosined grained soaps with 30 per cent. rosin can also be filled by crutching in about 30 per cent. filling material.

The filled soaps are ladled into smaller frames and, when cold, cut and dried for some time, whereby they gain considerably in firmness. They are then generally cut into square cakes, pressed, and brought into commerce.

Finally the filling of settled grained soaps with or without the addition of rosin may be mentioned. Soaps prepared from palm-kernel or cocoanut oil absorb but little filling material, but more of it can be incorporated if a larger per cent. of tallow or palm oil has been used for stock. The main point is that the soaps to be filled lie pure and bright in the kettle, show no lye-sharpness and are sufficiently firm. The soap is allowed to stand about 24 to 36 hours in the covered kettle to settle, and is then brought into the frame, leaving only room for the filling material, the latter being crutched in after a test has been made with a smaller quantity of the soap.

For filling settled grained soaps the above-mentioned filling of silicate of soda, soda solution and talc may be used, or one prepared by dissolving 1 part crystal soda in 2 to 3 parts silicate of soda of 38° B. Silicate of soda reduced to 28° B. with lye of 20° B. may also be used, as well as a composition of 103 parts silicate of soda, 19 parts soda lye of 22° B., 2 parts potash lye of 10° B., 7 parts potassium chloride and 5 parts crystal soda. Of the latter filling about 10 to 15 pounds for every 100 pounds of soap in the frame may be crutched in. It is always advisable to make a preliminary test so that if the silicate of soda should, contrary to expectation, crystallize, some lye of 38° B. may be added till the soap remains smooth after the filling has been added. When this has been ascertained the soap in the frame is filled.

Another filling material is prepared by dissolving equal parts of potash, soda ash and common salt in water so that the solution shows a density of 24° B. Before use the solution should be heated to about 176° F. As an especially good filling material may be mentioned the so-called caustic silicate of soda. It is prepared from 100 parts silicate of soda, 12½ parts caustic soda lye of 40° B., and 50 parts fresh lime-water (from 1 part lime and 5 parts water); the mixture is evaporated to a density of 45° B.

*Eschweg soap.*—The product known under this name may be classed as a semi-grained soap. It is still demanded by consumers in many localities with large mottle in a clear white ground and at the same time firm. Where no steam is available and fire only is used, or where the soap is brought to a boil with steam and finished with an open fire, the direct as well as indirect process of boiling is employed. Where steam is used or the soap is prepared from fatty acids by carbonate-saponification, the direct process of boiling is as a rule used.

Regarding the composition of soap-stock for Eschweg soap, it is best to have it consist of one-half palm-kernel oil and one-half animal fat or hard vegetable fat, such as bleached palm oil. If cocobanot oil is available, 30 per cent. of it

together with 70 per cent. of fats containing stearin suffices. In case palm-kernel oil is very dear, good soap may also be produced with 35 per cent. palm-kernel oil and 65 per cent. animal fats. Cotton seed or similar oil may also be used, but it should never exceed 20 per cent. of the stock. Hence the fats generally used for this kind of soap are: Palm-kernel oil, cheap cocoanut oil, tallow, bone fat, horse fat, lardaceous fats, further bleached palm oil, peanut oil and cotton-seed oil; the best stock, however, is as already mentioned, half palm-kernel oil and half tallowy fats.

If cotton-seed oil forms part of the stock without or very little animal fat or bleached palm oil, great care has to be exercised as cotton-seed oil soaps frequently make a great deal of trouble. Even if such soap was fine in boiling and lies faultlessly finished in the kettle, it may happen that in framing it, the mottle settles because the soap was still too hot. For this reason these soaps must be framed the colder the larger the frames are. It is advisable to always let them stand over night in the kettle, crutching them the next morning and then bringing them into the frames. Palm-kernel and cotton-seed oils, as well as old yellowish palm oils used for this purpose, and similar vegetable fats always keep the soap fluid for too long a time and the settling of the mottle later on is in consequence of it. When used for Eschweg soap cotton-seed oil always gives a smaller yield than animal fats, and even when it is cheap the profit is not so large as is sometimes supposed. Other fluid fats, such as olein, also give liquid soaps in which the mottle readily settles. Hence when such fluid fats are used as part of the stock, a perfect product can only be obtained by exercising great care.

It is generally supposed that the better qualities of soap which are firm when cut and are at once salable, as well as show a beautiful large mottle and a sufficiently uniform white ground, are prepared from better grades of fat than inferior qualities. The manufacturers of the latter often claim that their competitors work a good grade of tallow while they pro-

fess to use only bone fat and horse fat. This is however merely talk and amounts to nothing. If besides palm-kernel oil pure bone fats of a lighter color are available and the operation is properly conducted by not evaporating the soap too strongly, fitting it moderately and shortening it sufficiently, a product perfect in every respect can be obtained.

The formation of the mottle and the white ground serve, as previously mentioned, for the standard of the quality of Eschweg soap. The formation of the flux in the soap depends on the chemical composition of the fats, those which come here into question containing stearin and olein in varying proportions. When the soap has been properly made and the frame is covered, the firm stearin-soap crystallizes from the more fluid olein soap and thus the mottle appears when the soap, after cooling, is cut. The colored mottle is formed by the coloring matter passing into the more liquid olein soap. For the production of Eschweg soap with beautiful mottle and white ground, which is at once salable, the working of suitable fats and the choice of a proper method of boiling are not sufficient, the correct proportion of causticity of the soap also being a requisite for success; this will be referred to later on.

The question by which method of boiling—direct or indirect—a larger yield is obtained, cannot be answered off hand. Each method has its good points, the stock of fat used being also a factor. The skilled soap boiler will in any case obtain the same yield with either method, the differences rather depending on contingencies.

If the soap-stock contains a larger percentage of tallowy fats besides palm-kernel oil, some soap boilers, prefer indirect boiling, although it may be doubted that a larger yield is obtained. If on the other hand, the soap-stock contains much palm-kernel oil and cotton-seed oil and but little animal fat, most soap boilers prefer direct boiling, some of them claiming to have noticed that directly boiled soap has a better feel. The truth of the matter appears to be that the question of boiling is largely a matter of usage, each method giving about the

same yield. However, for rapid work, the direct method will have to be adopted, provided the fats used are clean. Dirty fats have to be preparatively boiled or previously purified.

For the preparation of Eschweg soap with the use of steam only, considerable experience is required to recognize the correct content of water and proper shortening of the soap. Still more attention and experience are required for the preparation of Eschweg soap with fine mottle and clear ground from fatty acid by carbonate-saponification.

Regarding the augmentation of Eschweg soaps it must first of all be mentioned that these soaps cannot be made with caustic soda lye alone, and hence it is impossible to draw a strict line as to where necessary additions stop and artificial augmentation commences. Every kind of Eschweg soap requires for its formation certain salts, either alkaline carbonates or silicates, or alkaline chlorides. An addition of silicate of soda can therefore be scarcely called an artificial augmentation, because by reason of the quantity of it absorbed by the soap, the latter will on an average require a smaller amount of other shortening agents.

A soap boiled chiefly from palm-kernel oil with perhaps a small quantity of cottonseed oil or other fluid or semi-fluid fats will only absorb an appreciable addition of silicate of soda with the use of very pure caustic lye, but a considerable quantity of other lighter salts, for instance, soda and potash. In consequence of this, with the use of the same stock of fat, such a soap treated with silicate of soda will give a smaller yield and present a poorer appearance than one shortened with other salts. The best results will always be obtained by taking part silicate of soda and using other salts for further shortening or filling. The reason for this lies in the nature of the soaps from these lean fats. They are too thin and cannot absorb such heavy additions without disturbing effect upon the formation of the mottle.

When fats containing stearin, bone fat, etc., are used, the treatment with silicate of soda is far less difficult, and in this

case it may even become a question of filling. With a soap-stock consisting of one-half palm-kernel oil and one-half bone fat or other tallowy fat, filling with silicate of soda up to 20 per cent., presents no difficulty whatever to the skilled soap-boiler, and he will still always have occasion to effect complete shortening with other salts, such as soda, potash or common salt solution. A few per cent. of talc may also be used without impeding the formation of the mottle. With the use of tallow in the above-mentioned soap-stock still more silicate of soda can easily be used for filling.

Soap moderately filled with silicate of soda has the advantage over one not thus treated of better retaining a smooth, pleasing exterior and possessing a better feel. If, however, too much silicate of soda has been used the soap, when stored for some time, becomes hard as stone and presents a bad appearance.

*Eschweg soap by the indirect way.*—The various methods of boiling Eschweg soap will here be described, but it must be borne in mind that it is impossible to give in a book such explicit directions as will be applicable to every case in practice. For the better understanding, a soap-stock will be given as an example, and the boiling of Eschweg soap by the indirect way first described.

*Stock:* Palm-kernel oil, 1000 pounds; bone fat, 500 pounds; horse fat, 500 pounds; caustic soda lye of 25° B., 2000 pounds; silicate of soda, 400 pounds.

For the conversion of the tallowy fats into grained soap, pure caustic soda lye of 18° to 25° B. is used. Bring the horse fat and bone fat into the kettle. For their saponification about 1000 pounds of caustic soda lye of 25° B. are required. To bring about rapid combination bring into the kettle together with the fat 400 pounds of lye and 600 pounds of water, start the fire or boil with direct steam till combination has been effected. When such is the case add gradually, keeping the mass constantly boiling, the rest of the lye and eventually 300 to 400 pounds of water until a clear, well-fitted paste boils thoroughly combined in the kettle. Continue boiling for half

an hour longer, and if the faint biting sensation produced upon the tongue by a sample remains unabated, one may be sure that all the fat has been thoroughly saponified and the paste can be salted out with about 7 to 8 per cent. of common salt until the lye runs off clear, and a fine, smooth grain boils lightly high in the kettle, this being the best indication of the spent lye settling well and readily. If too much salt has been used and a distended grain remains in the kettle, the spent lye settles badly and disturbances readily occur later on in boiling to a finish. The kettle is then covered and allowed to stand, best over night, for the spent lye to settle.

In the meanwhile scrap is melted in another kettle upon 1000 pounds of caustic soda lye of 25° B., which are required for the saponification of the 1000 pounds of palm-kernel oil and the 20 per cent. of silicate of soda is added. From the 1000 pounds of animal fats boiled to grain about 1600 pounds of moist grain are obtained. In order to here obtain also 200 per cent. yield, 400 pounds of water have to be added to this grain. Now add 900 pounds of the 1000 pounds of palm-kernel oil, keeping the remaining 100 pounds for fitting next day, cover the kettle and let it stand till the next day.

The fire is then started, and when the mass boils the grain is introduced. Combination is effected by means of the crutch and eventually with some direct steam, the latter being shut off when combination is complete. When the combined soap is thoroughly boiled through, it will boil, well and thick, high in the kettle. Fitting has to be controlled and a faint biting sensation should be perceptible when a sample is tested with the tongue. If the soap is found too strong it is fitted, as above stated, with the remaining palm-kernel oil. The solution of coloring matter is now added and when this has been incorporated by boiling, the soap is somewhat more shortened with brine of 24° B., a few per cent. being, as a rule, still required. This boiling to a finish is executed as follows: When fitting with the palm-kernel oil held in reserve is properly done and the solution of coloring matter incorporated, it can be readily

judged whether the soap has the right proportion of causticity ; if the soap easily boils high, is bright and breaks into roses over the entire surface of the kettle, it may be assumed that the correct quantity of alkali has been used. The soap may be considered finished when samples taken from the kettle lie without bubbles, and pieces the size of a dollar for five minutes remain in the center so soft that on pressing with the finger liquid soap still exudes. In this case the content of water is also correct, which may also be determined by the pressure-test with the finger, the soap not becoming directly hard like grained soap, but only gradually so. The soap should fall short from the paddle and form short curved points which become immediately cold. If, on the other hand, the soap is still viscid and leathery, it is yet too caustic and has to be carefully shortened with brine till it shows the above-mentioned indications of being finished.

*Eschweg soap by the direct way.* The preparation of Eschweg soap is much simplified by direct boiling, this method being actually much easier than the indirect process. By taking the previously-mentioned soap-stock as a basis or using somewhat more palm-kernel oil or in addition some cottonseed oil, the process remains the same.

Suppose, for instance, the stock consists of, palm-kernel oil 1000 pounds, bone fat 600 pounds, bleached palm oil 200 pounds, cottonseed oil 200 pounds, caustic soda lye of 25° B. 2000 pounds, silicate of soda 400 pounds. Boiling is executed as follows : The scrap is melted upon the 2000 pounds of lye, the silicate of soda is then added, and the hole then allowed to boil thoroughly. The stock of fat may be added in pieces, which melt in the hot soap, but it is better to introduce it in a liquid state. When all the fats with the exception of 100 pounds of palm-kernel oil, which are retained for fitting later on, are in the kettle, the mass is combined by crutching and with a moderate fire, the latter being increased only when a quite uniform soap has been formed. Fitting is also effected with the palm-kernel oil kept in reserve so that a moderately

biting sensation is perceptible when a sample is tested with the tongue. The soap is then colored, boiled free from froth and shortened in the same manner as previously described for the indirect process.

The use of a large percentage of cottonseed oil, or of only palm-kernel oil and cottonseed oil, for instance, 70 per cent. palm-kernel oil and 30 per cent. cottonseed oil, renders the preparation of Eschweg soap extremely difficult, not the boiling itself, but the finishing. It is best to fill such soap not at all with silicate of soda, or at the utmost with only 5 per cent. Generally speaking, this soap will not stand strong shortening agents, it being best to use brine of 15° to 18° B. or soda solution. It is advisable to allow the soap to cool over night and then to crutch it before bringing it into the frame; it is also well to cover the frame, especially if large.

*Boiling Eschweg soap with steam.*—This process requires considerable experience and everything has to be carefully adjusted so that, when the soap is boiled together, not many corrections have to be made. The operation of boiling so as to obtain a yield of 210 to 220 per cent. will be described below. The following soap-stock may serve as an example: Palm-kernel oil, 1000 pounds; tallowy fats, 1000 pounds; caustic soda lye of 30° B., 1600 pounds; silicate of soda, 400 pounds; brine of 24° B., 120 pounds.

The scrap is melted by steam upon the 1600 lbs. of lye of 30° B., and the stock of fat previously melted is immediately added, retaining, however, 100 lbs. of palm-kernel oil for fitting later on. It is also advisable to bring the silicate of soda into the kettle before adding the fat. Rapid and intimate combination is assisted by crutching and a thick, heavy soap will then immediately boil up, which is fitted with the palm-kernel oil held in reserve so as to produce a faint biting sensation upon the tongue. Now add carefully the 120 lbs. of brine, or at first only 80 lbs., and wait until it has been incorporated. Control again fitting, content of water and shortening. With a soap boiling conformable to proportions the

indications of its being finished are as follows: The soap, as previously mentioned should produce only a faint biting sensation upon the tongue. The content of water of the soap is noticed on the lower surface of a cold sample on a glass plate; if near the edge of the lower surface of the sample white stains have been formed, the content of water is correct. If, on the other hand, the entire surface is covered with white stains the soap is low in water and some water may be added. However, this does never happen if the directions given for boiling have been followed, since during this process about 10 per cent. of water passes with the steam into the soap, and in most every case a yield of at least 215 per cent., and generally somewhat more, is obtained. The content of water may also be correctly judged, after properly fitting, by the pressure-test with the finger; the soap should only gradually stand a firm pressure. The proper content of water is also indicated by the soap boiling thick and woolly. The proper proportion of causticity is shown by allowing a sample the size of a dollar to lie for five to six minutes upon a glass plate and then pressing it in the center with the finger when liquid soap should still exude. If the soap cools more rapidly some brine is wanting. From what has been said it will be seen that the whole operation has to be finished as rapidly as possible so that not too much water gets into the soap. The steam is finally shut off and after about two hours the soap is brought into the frame.

*Coloring Eschweg soap.* The coloring matters generally used are: For *grey*: Frankfort black or animal charcoal dust; for *red*: bole or ferric oxide; for *blue*: ultramarine by itself or mixed with Frankfort black. The coloring matter is stirred in water, some lye is added and the finished soap while boiling is colored with it until the tone of color shows itself plainly.

## CHAPTER XII.

### HARD SOAPS (CONTINUED).

#### II. PASTE SOAPS.

THE term paste-soap is frequently applied to such bar soaps as can be produced with a higher yield than grained soaps. However, products with a yield of 150 to 250 per cent. are generally called semi-grained soaps, to indicate that they possess a larger content of fat, and the mode of their preparation also gives rise to this designation because, by being more crunched together, an appearance more resembling that of grained soap is attained.

There is in some localities considerable demand for paste-soaps for household purposes, and their manufacture forms a remunerative branch of the soap industry. In fact, a soap factory, whether large or small, that does not make such soaps where there is a market for them, stands in its own light. Buyers will always be ready for them, because for ordinary uses they possess everything appreciated in a soap—they present a good appearance, lather readily, and are cheap. Even soaps with 250 to 400 per cent. yield are of fairly good quality, and cannot be called fraudulent because the filling incorporated with them partially possesses good detergent power.

The percentage of yield in the manufacture of these soaps varies very much, and the fact that some of them turn out unsalable is frequently owing to the disregard of the limit. There are certain conditions for the different yields which have to be observed and taken into account. If, for instance, it is calculated from the outset that at prevailing prices a yield of 300 per cent. of any kind of paste soap will pay, the

stock of fat and the lye will be nearly the same as for boiled semi-grained soap. If, however, in the process of manufacture, too much lye should be added at the start, the resulting soap would be defective. It would be too sharp, inclined to efflorescence, and dry out, because it lacks firm combination. If the defect to which excessive sharpness is due is detected before the soap is brought into the frame, it can be removed and compensated by the addition of fat. Water or aqueous common salt solution must of course also be added to prevent the soap from becoming poor in water. Paste soap poor in water sets out a frothy, more or less thick grain, and is then short. In such a case water has to be crutched in to fix the liberated salts.

With the application of good methods the manufacture of paste soaps is quite easy, but difficult with bad methods. The inexperienced soap boiler frequently uses too strong caustic lyes for combination and in consequence of this no union is attained. By long-continued firing the mass sometimes becomes too hot and is frequently brought to boiling whereby water constantly evaporates so that finally union cannot be attained. Paste soaps should never be brought to a boil, and the strength of the saponifying lyes should never exceed 35° B. Union is then readily attained and the soap will soon be finished. Somewhat stronger lye for saponification can only be used for silicate of soda soaps which are made from cocoanut oil. This has not been taken into consideration in some directions which have been published and, owing to this omission, complaints are made that the methods recommended are bad and good for nothing.

In the manufacture of paste soaps, as well as of every other kind of soap, allowance has to be made for the contingency of reducing and adding, because it is impossible to write directions so that they will be just right for every case. This is partly due to the caustic lyes, they being sometimes stronger and sometimes weaker, and also to the fact that the hydrometers do not always agree. Every trifle changes the

total result, and for this reason the soap boiler should be able to recognize an error which has been made, and know how to correct it.

In the hot state, paste soaps when finished, have the appearance of a clear paste solution and, when cold, yield a fairly firm product. The more water the soap contains and consequently the larger the yield is, the softer the product will be. In crutting a somewhat watery froth is always formed which, however, is not indicative of a defective soap. Froth is also formed in pouring the soap into the frame; this also does not matter as it generally disappears. At a temperature of 167° F. the soap purifies itself by ejecting all foreign particles.

According to their appearance paste soaps may be divided into two groups, viz., smooth and mottled soaps. As regards other qualities there are so many varieties that their number cannot even be approximately determined. The differences depend, on the one hand, on the yields which may vary from 180 or 250, up to 1600 per cent., and, on the other, on the many variations in color and shades.

All paste soaps can be prepared in the direct way and, in fact, this is the only correct method, but the lyes used for saponification must not be too strong. The lye and all the ingredients should be carefully weighed; this, however, is not necessary with silicate of soda soaps when caustic lye of a higher degree than is absolutely necessary for saponification is used, as then the silicate of soda does not settle and lumps are not formed. When the soap is finished the excess of lye is corrected by the addition of cocoanut oil. It is, however, different with soaps of low yield which to some extent are to resemble grained soap. In this case only so much caustic soda lye has to be used as is required for the saponification of the fats, and the filling agents should be applied in as concentrated a form as possible.

Generally speaking, it is best to use the following filling agents: Carbonate lyes, silicate of soda, and brine, the manu-

facture being thereby most readily effected. Besides the mentioned salt solutions, flour and more particularly talc are also employed, but the resulting soap presents, as a rule, a dull and dead appearance. Paste soaps are cheap products, but notwithstanding this they should as much as possible replace better qualities.

*Paste-soaps with a yield of 220 to 250 per cent.* Paste soaps with a yield of up to 275 per cent. should resemble grained soap and their manufacture differs from those with larger yields. If the product is to be an imitation of grained soap it must resemble the latter in its chief properties, dry out but little and be economical in use; this depends chiefly on the fats and the filling used.

*White soap with a yield of 220 per cent.* Palm-kernel oil or cocoanut oil 200 lbs., tallow or lardaceous fat 40 lbs., caustic soda lye of 30° B. 205 lbs., silicate of soda 65 lbs., water 10 lbs., brine of 24° B. 9 lbs.

*Yellow-white soap with a yield of 250 per cent.* Palm-kernel oil 200 lbs., rosin 40 lbs., caustic soda lye of 30° B. 204 lbs., water 10 lbs., silicate of soda 65 lbs., brine of 24° B. 9 lbs.

*Yellow soap with a yield of 275 per cent.* Palm-kernel oil 200 lbs., rosin 40 lbs., caustic soda of 30° B. 204 lbs., water 10 lbs., silicate of soda 110 lbs., brine 9 lbs.

Bring the fat or the fat and comminuted rosin into the kettle, start the fire, and melt the fat, whilst frequently crutching, without taking into consideration the melting of the rosin. When the temperature is about 167° F., crutch in about two-thirds of the lye without, however, aiming at combination. Next crutch in the water and then the silicate of soda, if there is any scrap, add it also, subsequent combination being thereby much promoted. If no scrap is available, cover the kettle for half an hour and keep up a moderate fire. Then remove the cover from the kettle, increase the fire, and commence crutching the soap together. The fat still present gradually disappears more and more, and passes into combination, until finally, with further crutching,

the soap reaches a stage when it is thought that complete combination may take place at any moment. Now withdraw the fire and continue crutting until complete combination suddenly takes place. Then at once crutch in the remainder of the lye and the silicate of soda, and finally the brine, which finishes the soap. Small corrections may now be made. If the soap has turned out right there will be in the kettle a fine dark and clear paste, which should produce quite a biting sensation upon the tongue and show good pressure under the finger. Soap too strong produces a very biting sensation upon the tongue and is glass-hard when pressed with the finger; soap too weak, besides being quite viscid, produces only a very faint biting sensation upon the tongue or none at all, and shows but little pressure under the finger. In the first case crutch in some oil, and in the other some lye. If the ingredients have been accurately weighed out the soap will, as a rule, be right and only trifling corrections be found necessary. The soap is frequently crutched and brought as cold as possible into frames holding from two hundred to twelve hundred pounds, in which it is also frequently crutched. The smaller the frames the finer the soap will be because mottle will be formed in larger frames if the soap is not sufficiently cold when brought into them.

In case scrap is worked, water has to be added in melting it according to whether it is more or less dried out, to prevent the soap from becoming low in water. Soaps low in water throw up a froth resembling grained soap, and in such a case water has to be crutched in till the froth disappears. These soaps must never be allowed to come to a boil.

For soaps the yield of which is to resemble grained soap, silicate of soda has to be used for filling and the process of manufacture as above described followed. With the use of potash solution and brine only, soaps smooth and less resembling grained soap are obtained as shown by the following compositions. These soaps should never be heated to above 185° F.

*Paste soaps with a yield of 250 to 275 per cent. and of smooth appearance.*

*Stocks.* I. Cocoanut oil or palm-kernel oil 90 lbs., tallow 10 lbs., caustic soda lye of 30° B. 84 lbs., potash solution of 30° B. 55 lbs., brine of 24° B. 36 lbs.

II. Cocoanut oil 85 lbs., tallow 15 lbs., caustic soda lye of 30° B. 84 lbs., potash solution of 30° B. 30 lbs., silicate of soda 30 lbs., brine of 23° B. 20 lbs.

III. Palm-kernel oil 90 lbs., tallow 10 lbs., caustic soda lye of 30° B. 84 lbs., potash solution of 30° B. 50 lbs., silicate of soda 35 lbs.

Mix the caustic soda lye and potash solution. Bring the fats into the kettle, melt them, and at 167° F. crutch in the lye, when combination will immediately result. When thorough combination is established crutch in the other fillings, the brine always last of all. When larger quantities are manufactured cover the kettle over night. The next morning remove any skin of froth which may have formed, scratch, eventually perfume, and bring the soap at a temperature of 158° to 167° F. into the frames.

*Paste soaps with a yield of 300 to 350 per cent.*

*Stocks.* I. Palm-kernel or cocoanut oil 100 lbs., caustic soda lye of 30° B. 84 lbs., potash solution of 30° B. 100 lbs. brine of 24° B. 46 lbs., or,

II. Palm-kernel or cocoanut oil 100 lbs., caustic soda lye of 30° B. 84 lbs., potash solution of 30° B. 80 lbs., silicate of soda 30 lbs., brine of 24° B. 15 lbs.

These soaps are made in the same way as the paste soaps previously described except that, when finished and thoroughly combined, they are allowed to stand for two hours in the well-covered kettle to insure intimate union. A sample of 2 to 4 ozs. is then taken and allowed to cool in a saucer when it should be quite firm. Should such not be the case, add from 5 to 10 lbs. of brine of 24° B., but such additional hardening will be but seldom required. In summer hardening may also be effected by the addition of some caustic soda

lye of 30° B., provided the soap does not show too strong a touch. In this case add hot water to the soap and scrutch thoroughly until good combination is effected. This defect, however, only occurs in very rare cases as the soap very readily combines immediately when lye and fat are crutched together, and it has reached a temperature of 188° F.

If the finished soap has a grain-like froth, want of water is generally the cause and this defect occurs, as a rule, only when scrap very much dried out is worked up. When scrap is used it should be added after the fat has been crutched together with the lye, adding at the same time 10 lbs. of water for every 100 lbs. of scrap but slightly dried out; and 20 to 25 lbs. for every 100 lbs. of very dry scrap.

*Paste soaps with 400 to 800 per cent. yield.*

*Stocks:* I. Cocoanut oil 100 lbs., caustic soda lye of 30° B. 84 lbs., potash solution of 30° B. 100 lbs., silicate of soda 50 lbs., brine of 22° B. 100 lbs.; or

II. Cocoanut oil 100 lbs., caustic soda lye of 30° B. 84 lbs., potash solution of 35° B. 300 lbs., silicate of soda 200 lbs., brine of 23° B. 100 lbs., water 30 lbs.

The soap with a yield of 400 per cent. from stock I is made in the same way as the paste-soaps previously described. In making soap with a yield of 800 per cent. from stock II, the brine has to be very carefully crutched in, it being advisable that the soap, after the soda lye and potash solution are combined with the fat, be again brought to 188° F. before crutting in the silicate of soda, and continue thus till everything is added. Should the soap not be sufficiently firm, it is hardened by the addition of lye of 30° B.

*Silicate of soda soap for washing in sea water.* A soap which contains a large quantity of silicate of soda can be used for washing in sea water or the hardest kind of well water. These readily soluble soaps are therefore much used on board of sea-going vessels.

*Silicate of soda soap with a yield of 320 per cent.* Cocoanut oil 100 lbs., caustic soda lye of 30° B. 100 lbs., potash solution of 30° B. 30 lbs., silicate of soda 100 lbs., water 25 lbs.

Bring the cocoanut oil together with the potash solution into the kettle and allow it to melt whilst stirring thoroughly. When the mass has reached a temperature of 188° F. crutch in the lye; when intimate combination has been effected bring the soap again to 188° F. and then carefully crutch in the silicate of soda, a large scoopful or small bucketful at a time. If lumps should form, crutch in 10 pounds more of caustic soda lye of 30° B. When the silicate of soda has been crutched in, add the water and withdraw the fire.

Allow the soap to stand quietly for two hours, or larger quantities over night, when the soap should produce a moderately biting sensation upon the tongue and lie free from froth in the kettle. In case the soap shows a strong touch and a glass-hard pressure, add a few pounds of fat and 10 to 15 pounds of water and crutch thoroughly, the fitting being thus regulated till the soap shows a moderate touch and good pressure. Viscidity of the soap is an indication of its having too little lye. If the soap produces a normal biting sensation upon the tongue and has much froth, water alone is wanting. Scrap to be worked up is at the outset brought into the kettle together with the required quantity of water, about 10 pounds of it for every 100 pounds of scrap but little dried out, or it may be added after the silicate of soda has been crutched in, and melted with a moderate fire whilst being frequently crutched. To very dry scrap 20 to 25 pounds of water must be added for every 100 pounds of it. The finished soap is allowed to cool to 167° F., and then brought best into iron frames of 12 to 30 cwt. capacity.

*Rosined paste soaps.*—As in the case of paste soaps without the addition of rosin, which have been above mentioned, those with the addition of rosin also require good stocks and caustic lyes not too strong. All these paste-soaps, whether they give large or small yields, or are made without or with the addition of rosin, must have one thing in common, namely, a soap-mass thoroughly combined in lye and fat and rosin, which exactly forms a paste suitable for filling. In the preparation of

rosined paste soaps double care has to be had that only lyes as caustic as possible reach saponification such as are capable of absorbing the filling without detriment.

The manufacture of rosined paste soaps is extensively carried on most everywhere, the varieties chiefly brought into commerce being of a yellow and brown color, and also partially of a black one. In preparing these soaps, no matter what their color may be, it must be borne in mind that rosin makes them soft, and, therefore, in most formulas the quantity by weight of rosin which can be used is given. Only good fats can be employed for rosined paste soaps, and, if possible, some tallow, besides palm-kernel oil or cocoanut oil and crude palm oil. Soaps with a very large content of rosin can then be made, which, however, will not stand much filling.

Such soaps with a large content of rosin may consist of the following stock: Palm-kernel oil 150 lbs., tallow 60 lbs., bone-fat or crude palm oil 40 lbs., pale rosin 250 lbs., caustic soda lye of 30° B. 420 lbs., silicate of soda 50 lbs., soda crystals 50 lbs.

Serviceable soaps could be obtained by omitting the soda crystals and silicate of soda from the stock, but their employment will result in a product that is smoother, and feels drier and firmer. The simplest method of preparing such soaps is to crutch the lye to the rosined fat and effect thorough combination by subsequent heating.

Heat the fat to about 180° to 194° F., and dissolve in it the comminuted rosin. Allow the mass to cool to 167° F., and then slowly crutch in the lye which contains the silicate of soda, till all is thoroughly combined. A moderate fire may be kept up but 190° F. should not be exceeded. Combination may be promoted by warming the lye and adding any available scrap to the rosined fat. The mass thickens as soon as it becomes too cold and a moderate fire should therefore be kept up. When thorough combination has been effected add the soda crystals and allow them to melt, crutching frequently. The fire is then withdrawn and the soap allowed to stand

quietly for two hours, so that thoroughly intimate combination results. It is then well crutched and brought into frames holding from 12 to 15 cwts., and again crutched somewhat cold in them.

*Rosined paste soap with a yield of 300 to 400 per cent.* Stocks may be composed as follows:

I. Cocoanut oil 100 lbs., palm-kernel oil 80 lbs., crude palm oil 20 lbs., rosin 30 lbs., caustic soda lye of 30° B. 198 lbs., potash solution of 30° B. 110 lbs., brine of 22° B. 110 lbs., silicate of soda 65 lbs., or .

II. Palm-kernel or cocoanut oil 100 lbs., crude palm oil 5 lbs., rosin 10 lbs., caustic soda lye of 30° B. 96 lbs., potash solution of 30° B. 55 lbs., brine of 22° B. 40 lbs., silicate of soda 15 lbs.

Dissolve the rosin in the fat, then crutch in the potash solution and immediately afterwards the caustic soda lye, combination being readily attained in this manner. When combination is complete raise the temperature of the soap to 190° F. and crutch in the brine. Now remove the fire and let the kettle stand quietly for one hour to allow intimate combination to take place, and then crutch in the silicate of soda. Then take samples. If they are not firm enough, harden, in summer as well as in winter, by the addition of a few pounds of soda crystals and crutch until they are melted; then bring the soap into frames.

*Rosined paste-soap without brine.* Stock: Cocoanut oil 100 lbs., crude palm oil 10 lbs., pale rosin 25 lbs., caustic soda lye of 20° B. 200 lbs., silicate of soda 75 lbs., soda crystals 30 lbs.

This stock yields a cheap soap of fine quality which by reason of the low-grade lye used, is easy of manufacture. Dissolve the rosin in the fat, gradually crutch in the lye, bring the soap to 190° F., crutch in the silicate of soda, and immediately after that add the soda crystals. Then withdraw the fire, crutch several times till the soda crystals are dissolved, and bring the soap into the frames. If scrap is available add it after the lye has been crutched in. Should it hap-

pen that the soap turns out somewhat soft, harden it by adding 1 to 2 lbs. of caustic soda lye of  $36^{\circ}$  B.

*Transparent rosined paste-soap.* A transparent paste-soap of fine appearance the manufacture of which is simple and easy, is obtained, when properly made, from the following stock : Cocoanut oil 240 lbs., crude palm oil 100 lbs., rosin 30 lbs., potash lye of  $26^{\circ}$  B. 190 lbs., caustic soda lye of  $26^{\circ}$  B. 190 lbs., potash solution of  $35^{\circ}$  B. 80 lbs., brine of  $22^{\circ}$  B. 208 lbs., silicate of soda 160 lbs., soda crystals 60 lbs.

Dissolve the comminuted rosin in the melted oil and fat, crutch in the potash lye and when combination has taken place, crutch in the soda lye and immediately after that the potash solution. Bring the soap to  $190^{\circ}$  F., crutch in the silicate of soda, then add any scrap available and the soda crystals, keeping up a moderate fire until all is dissolved. Then withdraw the fire, allow the soap to cool to  $167^{\circ}$  F., and bring it into the frames.

*Rosined paste-soap with talc.* Stock : Cocoanut oil, 250 lbs. ; crude palm oil, 200 lbs., caustic soda lye of  $25^{\circ}$  B., 570 lbs.; rosin, 100 lbs.

*Filling :* Talc, 270 lbs. : water, 500 lbs. ; silicate of soda, 100 lbs. ; soda crystals, 50 lbs.

Bring the rosin and cocoanut oil into the kettle, boil them with the lye to paste, then add at once the palm oil, and boil the whole thoroughly. The entire stock of fat may also be at once brought into the kettle and saponified with the lye of  $25^{\circ}$  B., but care must be had that the soap does not become thick, as in that case it would not have sufficient fitting. To avoid this defect it is advisable to keep 50 pounds of the crude palm oil in reserve and add them later on. In the meanwhile the talc has been stirred together with the water. When saponification has taken place add this mixture and combin it intimately by vigorous stirring so that the talc does no scorch too much. When everything is thoroughly combined add the soda crystals and, when they have also been incorporated, the palm oil kept in reserve. When this is also thor-

oughly combined, regulate the fitting and firmness of the soap. It should produce quite a biting sensation upon the tongue and show good finger-pressure. If it does not produce a biting sensation upon the tongue, or only a very faint one, and shows no pressure, lye is wanting. Add lye of 25° B. and boil moderately till the result desired has been attained. Then remove the fire and stir vigorously to prevent scorching. When the soap is quiet after boiling, crutch it frequently and, if there is a large quantity of it, allow it to stand over night to cool in the kettle. The next morning crutch any tale which may have separated into the soap, bring the latter into small frames and crutch till it can no longer settle.

*Alkaline silicate or water-glass compositions* are most suitably prepared from cocoanut oil. Water-glass or soluble glass—sodium silicate or potassium silicate—is used in soft soaps as well as in hard soaps, but to the greatest extent in these water-glass compositions, because of all watery filling agents it dries in the least and also possesses good detergent power. It makes hard water soft and prevents the soap from coagulating in it. A few good processes are here given. As stocks serve :—

I. Cocoanut oil 100 lbs., caustic soda lye of 25° B. 100 lbs. mixed with water 70 lbs., water-glass 120 lbs. mixed with caustic soda lye of 25° B.

The cocoanut oil is crutched together with the caustic soda lye at about 194° F. and brought into combination, when the water-glass is immediately crutched in and the soap is finished. If desired some soda crystals may also be added.

II. Cocoanut oil 100 lbs., caustic soda lye of 25° B. 100 lbs. mixed with water 50 lbs., water-glass 150 lbs. mixed with caustic lye of 25° B. 15 lbs., potash solution of 30° B. 50 lbs.

Bring the oil into the kettle and boil it with the lye to a paste by steam or over an open fire, and then add immediately the potash solution. Now remove the fire and crutch in the water-glass. Should it happen that lumps are formed in crutting in the water-glass, crutch very slowly, and

eventually use fire or steam. Any available scrap may from the start be brought into the kettle.

*Mottled soaps.*—Notwithstanding their good appearance and great similarity to Eschweg soap, mottled soaps, by reason of their large yield have to be classed with paste-soaps. Generally speaking most of them could formerly be made with a yield of less than 320 per cent. Many soap boilers tried to improve them in so far as to make their content of fat approximate that of Eschweg soap, but, as a rule, it was lost labor. The experiments always failed on account of the large content of salt required by these soaps for fixing the mottle. The best varieties, when freshly cut, have a yield of 320 to 340 per cent., and the limit of the largest yield is about 700 per cent. The better varieties can be considerably improved so that they become more resistant in use by placing them, in the form of blocks not too large, or better in the shape of slabs, for a few days in dry rooms. They lose in an incredibly short time about 20 per cent. in weight. However, drying should not be carried that far, because the soap then develops its worst quality, namely, the salts which have been more concentrated by crutching, crystallize out.

The manufacture of mottled soap was originally considered a secret, but it is of course no longer so, and at present soaps with a yield of 250 to 260 per cent. can be produced, considerable experience being, however, required for this. The manufacture of these soaps is quite easy if the soap boiler knows on what the formation of mottle is based. This depends on the fitting; the soap should be so fitted that the water-glass slowly crystallizes out with the color. If the basis soap is too weak in the fitting, the water-glass color crystallizes out too quickly and settles on the bottom. With soap too strongly fitted the color solution can crystallize out but little or not at all. Hence the first requirement is to make a correct basis-soap. It must be properly constituted for the formation of mottle and capable of holding it as much as possible. How the mottle is to be formed in the soap, whether large or small,

is a matter of experience. Another factor of as much importance as the knowledge of the proper mottle, deserves careful consideration. Mottled soap should never be poor in water; if such be the case the result will always be a poor and small mottle, no matter how well the principle of mottling may be understood and applied. Deficiency in water is the greatest defect in all kinds of paste soap, and more particularly so in mottled products. When water is wanting, the salts by being liberated produce a severing effect upon the combination of the soap, and a fine mottle cannot be produced. Deficiency in water is a defect which can be readily determined and just as readily removed, but, nevertheless, many soap-boilers proceed in this case with the greatest timidity, being under the impression that too much water might be brought into the soap. Deficiency in water is indicated by the appearance on the surface of a thick, heavy froth, which is often grainy, and the thicker and heavier and more grainy it is the poorer in water the soap is. If the froth is grainy a large quantity of water is wanting, in fact, so much water that an inexperienced soapboiler hesitates to add as much as is required. There is no reason for being timid even after the addition of 100 or more pounds of water, the latter is wanting so long as the soap possesses this heavy froth. Mottled soaps should only have so much watery froth as is formed by crutching. The soap specially requires much water when scrap is worked with the stock; on this depends the success of the operation. It is no matter whether one formula prescribes caustic soda lye of 20 or 22° B., or another brine and potash solution of somewhat different degrees, or that in some directions such great value is set upon the color solution that special attention is called to the importance of working according to a correct percentage. These perplexing details make the beginner timid, and besides, many of these directions are of no value whatever. All that is necessary is to prepare a proper paste which becomes sufficiently firm, and to regulate the fitting so as to enable the water-glass color solution to

crystal out. Everything else is superfluous and only makes the operation more difficult.

The fats chiefly used for this kind of soap are cocoanut oil and palm-kernel oil, the latter being mostly employed and is more suitable for a yield of up to 400 per cent. than the former, which, by reason of its greater pasting capacity, should only be used in stock for larger yields; for lower yields it exerts a disturbing influence. Some tallow may be recommended as an additional fat, it possessing the property of making the soap congeal more rapidly in the frame after the formation of the mottle. Soap with a yield of up to 400 per cent. should be made with palm-kernel oil alone or eventually with the addition of some tallow or another fat. For a yield of from 450 to 550 per cent. the stock may consist of at least one-half cocoanut oil, and for a yield of over 600 per cent. cocoanut oil alone should be used as stock. For very large yields—from 500 per cent. up—the salt solutions for filling should be somewhat less concentrated than for lower yields.

Many soapboilers use for the thorough saponification of 100 per cent. fat 110 per cent. of caustic soda lye of 22° B. A more recent method, which is considered better, prescribes for 100 per cent. of fat 115 per cent. of caustic soda lye of 20° B. In some factories the soap while being saponified with caustic soda is allowed to boil and the solutions are then crutched in. However, the better way is not to allow the soap to boil, but to heat it only to 190° F. By boiling vigorously much water is evaporated, froth is formed, and the salts are liberated, which has a bad effect on the thorough combination of the soap. It is therefore more correct to bring the stock of fat into the kettle, saponifying it by crutting it together with the lye and at once crutting in the potash solutions, then heating the soap paste to 190° F., and finally crutting in the brine. The finished basis-soap should have pressure and be a paste-soap which spins well, *i. e.*, runs in threads from the paddle. For a test, take a sample with the paddle, wait half a minute, and let it run off, when it should spin well. Soap too hot, though

containing sufficient water, shows this spinning in a less degree. Potash solution and brine are the best fillings agents, others being less suitable. The use of much soda solution should especially be avoided, such filling being very detrimental, particularly to winter soaps, which then tremendously effloresce. All the salt solutions which are used cause disturbances when they are either too weak or too strong, the soap in the first case being rendered too soft, and in the other, grainy froth is formed similar to that when the soap is deficient in water. When a correct basis-soap has been prepared it is tested as to whether it will absorb the solution of coloring matter without crystallizing. This, however, will be referred to later on.

*Mottled soap with a yield of 260 per cent.*—The manufacture of mottled soap with such a low yield is, as previously stated, extremely difficult and requires considerable experience. The following stock may be used: Palm-kernel oil, 200 pounds, or palm-kernel oil, 180 pounds; tallow, 20 pounds; caustic soda lye of 20° B., 230 pounds; potash solution of 35° B., 40 pounds; brine of 24° B., 50 pounds.

*Color solution.*—Ultramarine,  $\frac{1}{2}$  pound, dissolved in water, 10 pounds; waterglass, 10 pounds; caustic soda lye, 2 pounds.

The chief difficulty with this soap is the formation of the mottle. Its manufacture is the same as that of mottled soaps with larger yield, which will be more particularly referred to later on. It may here only be said that this soap forms mottle with great difficulty, and as it contains less water and is consequently thicker, it has to be brought hotter into the frames and well covered. It is advisable to bring it at 190° F. into the frame and keep it well covered till the mottle is formed, when it is uncovered and allowed to cool.

*Mottled Soap with a yield of 350 per cent. Stock.*—Palm-kernel oil, 270 pounds; tallow, 30 pounds; caustic soda lye of 20° B., 345 pounds; potash solution of 35° B., 160 pounds; brine of 24° B., 215 pounds.

*Color solution:* Ultramarine  $\frac{1}{2}$  lb., water 10 lbs., caustic soda lye of 20° B. 2 lbs., water-glass 10 lbs.

*Mottled soap with a yield of 450 per cent.*—Palm-kernel oil 100 lbs., caustic soda lye of 20° B. 115 lbs., potash solution of 33° B. 105 lbs., brine of 22° B. 145 lbs.

*Mottled soap with a yield of 550 per cent.*—Palm-kernel oil 70 lbs., cocoanut oil 30 lbs., caustic soda lye of 20° B. 115 lbs., potash solution of 30° B. 150 lbs., brine of 21° B. 200 lbs.

*Mottled soap with a yield of 700 per cent.*—Cocoanut oil 90 lbs., tallow 10 lbs., caustic soda lye of 20° B. 115 lbs., potash solution of 30° B. 210 lbs., brine of 22° B. 280 lbs.

The color solution for the yields of 450 to 700 per cent. is as follows: Coloring matter 4 ozs., water 6 lbs., caustic soda lye of 20° B. 1 lb., water-glass 6 lbs.

Any of these stocks may be brought into the kettle without fear of meeting with difficulties. Start the fire and saponify the fat by gradually crutching in the lye. When good combination has been effected, crutch in the potash solution and bring the soap to 190° F. Then at once crutch in the brine and again bring the soap to 190° F. If scrap is available it may be added after crutching in the brine and melted with a moderate fire; according to whether the soap is less or more dried out, 10 to 20 pounds of water for every 100 pounds of scrap have to be added. Now withdraw the fire and cover the kettle for half an hour or so that the soap may still more intimately combine and the watery froth caused by crutching disappear. The small quantity of froth which still remains is skimmed off. From what has been said it will be seen that preparation of the basis-soap is quite simple and, if the work has been properly done, mottling is also a simple process. The soapboiler may now proceed to make the necessary tests. It is always well to reserve of the caustic lye of 20° B., which serves for saponification, 1 to 2 pounds for every 100 pounds of stock of fat to correct the soap in case it should be too weak. If, on the other hand, it is too strong, correction is more difficult, as one does never know how much oil has to be added. The lye used for the color solution is also taken from that kept in reserve.

Coloring the framed soap at hap-hazard or forcing it to mottle properly will, as a rule, turn out badly, because, even if all the ingredients have been most accurately weighed, the soap cannot be turned out so accurately that everything is at once in accord. The basis-soap used for making tests may have a temperature of 176° to 194° F.; it should not be colder than that, as otherwise the formation of mottle in the small testing vessels used becomes difficult. Bring into a scoop or small box 15 to 20 pounds of the soap in the kettle and stir in a small quantity of the color-solution. If it stirs in smoothly or crystallizes in separate minute dots, the soap has turned out quite right and may be colored in the kettle. If, however, the color-solution immediately on being stirred in crystallizes in larger patches, the basis-soap is still too weak and 2 to 3 lbs. of the lye kept in reserve have to be added to the color-solution. The test is repeated till the color-solution stirs in smoothly when the soap in the kettle is also colored. When the color-solution can be stirred in smoothly, the soap may have turned out right and can at once be colored in the kettle. The testing vessel should be well covered and allowed to stand quietly for one-half to three-quarters of an hour alongside the kettle containing the colored soap. If, after this time, a mottle is not noticed, the soap is too strong; in this case, crutch in 2 to 3 lbs. of oil and 6 to 7 lbs. of water, and the mottle will then result. If everything has been accurately weighed only trifling corrections will, as a rule, be required. If the soap has formed a faint scarcely perceptible mottle, it has turned out right and can at 167° F. be brought into the frame in which it remains covered till the mottle has set when it is uncovered. Should the soap show inclination towards allowing the mottle to settle, it may, without hesitation, be again crutched through and again covered for the formation of mottle. Such soap, as a rule, is slightly too weak, but this does not matter as it forms a large fine mottle, while that of soap more strongly fitted forms with greater difficulty and is smaller. Such soap shows no inclination towards allowing the mottle to settle.

If the soap after standing in the kettle for half an hour already forms a very large mottle, it is an indication that the basis-soap is somewhat too weak; in this case crutch in 1 to 2 lbs. of caustic soda lye of 20° B., and then bring the soap at 167° F. into the frame.

Small batches may be boiled in the morning and brought into the frame in the afternoon, but, as a rule, it is better to make all these kinds of soap in the afternoon and bring them into the frame the next morning. When the soap is finished up to the formation of mottle, cover the kettle till the next morning; then remove the cover, crutch through, and measure the temperature; if the latter is below 172° F., add 10 to 20 lbs. of water to the soap, start the fire, and bring the temperature to 172° F. Soaps which are covered hot at night, always allow the mottle to settle till the next morning, but this is not to be considered as a defect. The chief difficulty in the manufacture of these soaps lies in the formation of the mottle, but this is readily accomplished if the basis-soap has been accurately fitted and is not deficient in water.

## CHAPTER XIII.

### DOMESTIC SOAPS BY THE COLD PROCESS AND SEMI-WARM PROCESS.

*Fats and lyes for saponification by the cold process.*—By saponification by the cold process is understood the preparation of soaps—chiefly the cheaper kinds of toilet soaps, but also domestic soaps—by simply stirring together melted cocoanut or palm-kernel oil with the exact quantity of lye required for saturation. The process is based upon the property of the various kinds of cocoanut oil and of palm-kernel oil to enter with high-grade caustic lyes into a combination which by subsequent self-heating in the frame yields a firm white soap, particularly distinguished by an abundant lather. The capacity of the above-mentioned oils of readily saponifying is so great that in this process other fats and oils that saponify with difficulty can be used in connection with them. This fact is taken advantage of, and a certain percentage of tallow, palm oil, best quality of bone fat, horse fat, lard, oleic acid, cottonseed oil, peanut oil, sesame oil, olive oil and castor oil, as well as rosin, is frequently used in the manufacture of these soaps.

No matter whether the stock contains tallow and other fats and oil or not, the mode of preparing these soaps remains always the same, there being but slight deviations as regards the temperature of the fats to be saponified and the quantity of lye required for saponification. Concerning these two points it must be borne in mind that of all the fats and oils here in question, cocoanut oil requires the largest quantity of lye for its complete saponification. Next come tallow, lard and olive oil, about the same quantity of lye being consumed

by each of them. Castor oil requires the least lye. All the fats used for cold saponification should be as fresh and pure as possible, and above all, not be rancid, as otherwise the soap made from them might also turn rancid. As regards the second point, it may be laid down as a rule that the higher the melting point of a fat to be used for saponification by the cold process is, the higher the degrees of heat during the operation have to be kept. Thus, for instance, tallow, if it could be worked at all by itself, would have to be kept at  $140^{\circ}$  to  $149^{\circ}$  F., while if worked in conjunction with  $\frac{1}{2}$  to  $\frac{1}{3}$  cocoanut oil, a temperature of  $104^{\circ}$  to  $113^{\circ}$  F. would suffice in the one case, and one of  $90^{\circ}$  to  $95^{\circ}$  F. in the other.

The lyes for soaps by the cold process are best prepared from high-grade caustic soda. By using 200 lbs. of water for dissolving 100 lbs. of caustic soda, a lye of about  $40^{\circ}$  B. is obtained. It should be kept in a tightly closed holder, and is generally worked at a strength of  $36$  to  $38^{\circ}$  B. For one pound of cocoanut oil half a pound of caustic soda lye of  $38^{\circ}$  B. is estimated, such a mixture yielding a soap which is very firm when slabbed. Accurate weighing of fat and lye is a requisite for the success of the soap; if not enough lye is added the soap becomes soft and spongy, and if too much, hard and brittle, and even separates paste or lye.

For filling domestic soaps made by the cold process, silicate of soda, talc, filling lye, potash solution and brine are chiefly used. As the soap becomes much heated by the filling agents, it has to be brought into smaller shallow frames holding from 65 to 130 lbs. each, and allowed to stand in them uncovered, as otherwise oil readily deposits.

At present domestic soaps are also frequently made by the semi-warm process, by first allowing self-heating, eventually with the assistance of heat, to take place in the mass stirred together from fat and lye. After thorough crutching the paste grained soap formed, to which the filling has been added either previous to or after the appearance of self-heating, is brought into the frames. The same fats and lyes as

for soaps by the cold process are used and silicate of soda, filling lye, potash solution and brine are chiefly used for filling.

*Directions for domestic soaps by the cold process and semi-warm process.* Stock for prime quality of white domestic soap. Tallow 20 lbs., cocoanut or palm-kernel oil 30 lbs., caustic soda lye of 38° B. 25 lbs., potash solution of 20° B. 3 lbs., brine of 20° B. 2 lbs.

Stir into the melted fats at 95° F., the lye and when the mass begins to thicken add, whilst stirring constantly, the solutions one after the other, and bring the soap into the frames.

*Stocks for cheaper domestic soaps:* I. Cocoanut oil 50 lbs., palm-kernel oil 50 lbs., caustic soda lye of 23° B. 125 lbs., silicate of soda of 30° B. 25 lbs., 96 per cent. alcohol 9 to 11 ozs.

II. Cocoanut oil 50 lbs., palm-kernel oil 50 lbs., peanut oil 20 lbs., caustic soda lye of 23° B. 124 lbs., silicate of soda of 34° B. 25 lbs., 96 per cent. alcohol 9 to 11 ozs.

Stir the melted fat of stock I at about 99° F., and that of stock II at about 140° F., together with the lye and when combination has been effected stir in the silicate of soda previously heated to from 68° to 77° F. When the mass, whilst being thoroughly crutched, appears to be completely broken up, it is run into a frame and the 96 per cent. alcohol sprinkled over it, the soap being thereby immediately contracted. If it is to be colored or mottled the coloring matter must be ready to hand, as the soap rapidly congeals. For mottling, ultramarine or Frankfort black mixed with water is used.

According to a similar process a cheap soap is prepared from Ceylon cocoanut oil, 40 pounds; caustic soda lye of 32° B., 40 pounds; brine of 18° B., 40 pounds, and silicate of soda of 38° B., 50 pounds.

The melted cocoanut oil is at about 99° F. stirred together with 25 pounds of the lye, the remaining 15 pounds of the lye being poured into the brine, and this mixture of brine and lye

is then gradually added to the soap-mass, care being had that the latter remains in combination. The silicate of soda is then added in a thick jet so that the soap completely separates. Now crutch so that the whole is like a rice paste and no lumps are noticed. Then sprinkle 1 pint of 96 per cent. alcohol over the soap, crutch through and run the soap quickly into the frame, allowing the latter to stand uncovered.

Talc is worked together with the following stocks:

I. Ceylon cocoanut oil, 50 pounds; hog fat, 50 pounds, caustic soda lye of 36° B., 55 pounds; talc, 25 pounds.

II. Ceylon cocoanut oil, 65 pounds; peanut oil, 35 pounds; caustic soda lye of 35° B., 55 pounds; talc, 30 pounds.

Stir the talc together with the fat at about 122° F. and successively add the lye. When the soap thickens bring it into small frames.

A white soap (165 to 220 per cent. yield) can be prepared by the semi-warm process from the following stocks:

I. Palm-kernel oil, 85 pounds; tallow, 15 pounds (both fats at 113° F.); caustic soda lye at 38° B., 50 pounds; water, 3 pounds; potash solution of 20° B., 15 pounds.

II. Palm-kernel oil, 60 pounds; tallow, 40 pounds; caustic soda lye of 37° B., 55 pounds; solution of 20° B. of equal parts of sugar, common salt and potash in water, 25 pounds.

III. Palm-kernel oil 90 lbs., tallow 20 lbs., caustic soda lye of 30° B. 85 lbs., potash solution of 20° B. 15 lbs.

IV. Palm-kernel oil 80 lbs., tallow 20 lbs., caustic soda lye of 33° B. 80 lbs., silicate of soda of 38° B. 40 lbs.

V. Palm-kernel oil 90 lbs., tallow 10 lbs., caustic soda lye of 38° B. 50 lbs., water 3 lbs., silicate of soda of 38° B. 36 lbs., mixed with caustic soda lye of 38° B. 5 lbs., and potash solution of 25° B. 6 lbs.

VI. Palm-kernel oil 50 lbs., tallow 25 lbs., hog fat 25 lbs., bone fat 15 lbs. (the fats at 149° F.), caustic soda of 38° B. 64 lbs., silicate of soda of 38° B. 48 lbs., potash solution of 15° B. 17½ lbs.

The caustic soda lye, eventually mixed with the water, is

crutched into the fat at about  $122^{\circ}$  F., except when otherwise mentioned in the stocks, and then the filling. The pan is then covered for some time, being eventually moderately warmed and the mass occasionally crutched through. When self-heating has set in, the mass is stirred. When the soap on being tested is sufficiently firm and produces a faint biting sensation upon the tongue, it is brought into smaller frames. The stock of fat may also be saponified with the lye, and the filling, previously heated, crutched into the soap when thoroughly combined and bright.

*Rosined soaps.*—Two methods are chiefly employed for the manufacture of these soaps, the lye being either stirred together with the rosined fat, or vice versa, the rosined fat with the hot lye. Soap prepared by the first method is frequently somewhat sticky because saponification is not so intimate as by the other method. The above-mentioned fats are also used for making these soaps and for filling agents, chiefly talc, silicate of soda, potash solution, etc.

*Stock for rosined soap with talc.*—Tallow 42 lbs., palm-kernel oil 43 lbs., pale rosin 15 lbs., caustic soda lye of  $37^{\circ}$  B. 50 lbs., talc 5 to 7 lbs.

Melt the tallow and oil, dissolve the rosin in the melted mass, stir in the talc and strain. Crutch the lye into the rosined fat when cooled to about  $149^{\circ}$  F. The soap when thoroughly combined is brought into small frames.

*Stock for rosined soap with 50 per cent. rosin.*—Ceylon cocoanut oil 32 lbs., palm-kernel oil 16 lbs., pale rosin 24 lbs., caustic soda lye of  $37^{\circ}$  B. 36 lbs., soap-yellow about 28 ozs., dissolved in boiling water.

Dissolve the comminuted rosin in the hot melted fat. Allow the rosined fat to cool to about  $140^{\circ}$  F., add the color solution and then, stirring vigorously, the lye. The pan is then covered warm for about 20 minutes, when thorough combination will take place. The soap is then brought into the frame, stirred through short and lightly covered. After having been cut the soap is allowed to stand for a short time, which makes it not only firmer, but also more transparent on the edges.

*Stock for rosined soap with 100 per cent. rosin.*—Palm-kernel oil, 35 pounds; Ceylon cocoanut oil, 15 pounds; pale rosin, 50 pounds; caustic soda lye of 37° B., 50 pounds; soap-yellow, 1 ounce dissolved in 1 pound of boiling water.

Dissolve the rosin in the hot oil, allow to cool to 167° F., strain the rosined fat, add the color-solution, and then stir in the lye. After crutting somewhat the soap will come in quite a thick paste, which can be immediately brought into the frame in which it is allowed to stand uncovered.

Scrap from soaps made as above described is dissolved in the hot oil and thus utilized to advantage. If it is very much dried out, add some water.

By the semi-warm process a fine quality of rosined soaps may be prepared from the following stocks:

I. Palm-kernel oil, 80 pounds; Ceylon cocoanut oil, 20 pounds; rosin, 14 pounds; caustic soda lye of 37° B., 58 pounds; silicate of soda of 38° B., 40 pounds, mixed with caustic soda lye of 37° B., 7 pounds.

II. Palm-kernel oil 90 pounds, tallow 10 pounds, rosin 10 pounds, caustic soda lye of 36° B. 55 pounds, silicate of soda of 30° B. 30 pounds, mixed with caustic soda lye of 36° B. 4 pounds, and potash solution of 30° B. 5 pounds.

III. Palm-kernel oil 50 pounds, pale rosin 10 pounds, caustic soda lye of 30° B. 40 pounds, potash solution of 30° B. 7½ pounds.

IV. Palm-kernel oil 90 pounds, pale rosin 10 pounds, caustic soda lye of 33° B. 60 pounds, silicate of soda of 38° B. 40 pounds, mixed with caustic soda lye of 33° B. 3 pounds.

V. Palm-kernel oil 183 pounds, rosin 33 pounds, caustic soda lye of 37° B. 119 pounds, silicate of soda of 38° B. 64 pounds, potash solution of 30° B. 10 pounds.

Melt the oil and rosin over a moderate fire and dissolve in the melted mass, whilst constantly stirring, any scrap on hand. Crutch into the rosined fat, when cooled to about 167° F., the lye previously weighed and mixed, the silicate of soda and the potash solution. After again crutting through, cover the mass

for some time, and after the appearance of self-heating it will, when crutched, come to quite a liquid paste. When, on being tested, this paste shows pressure and produces a faint biting sensation upon the tongue, it is brought into the frames. Any corrections required can be readily made by the addition of warmed lye or liquid oil. If the soap is to have a pale yellow color dissolve for every 100 pounds of stock  $\frac{3}{4}$  to  $1\frac{1}{2}$  ounces of soap-yellow dissolved in 1 pound of boiling water, and add this solution to the rosined fat previously to stirring in the lye-mixture.

*Transparent rosin soap* may be prepared from Ceylon cocoanut oil 50 pounds, crude palm oil  $12\frac{1}{2}$  pounds, pale rosin  $37\frac{1}{2}$  pounds, caustic soda lye of  $38^{\circ}$  B. 50 pounds, water  $2\frac{1}{2}$  pounds, and about 8 ounces of 96 per cent. alcohol.

Melt the cocoanut oil and rosin over a moderate fire, then add the palm oil and let that also melt. The melted rosined fat is strained and when it shows a temperature of about  $176^{\circ}$  F., stir in the lye in a fine jet. When combination has been effected, add the water to the thick soap-mass, the latter becoming somewhat more liquid by this addition. Now crutch in the alcohol and allow the soap to rest for about one hour, the pan being well covered, in order to bring about a more intimate union of the mass. The somewhat thick, transparent soap is then brought into the frame, again drawn through with the crutch and allowed to stand without being covered.

*Stock for a pale rosined soap*.—Ceylon cocoanut oil 160 lbs., tallow 30 lbs., hog fat 10 lbs., pale rosin 50 lbs., Venetian turpentine 5 lbs., caustic soda lye of  $36^{\circ}$  B. 127 lbs., potash solution of  $30^{\circ}$  B.  $12\frac{1}{2}$  lbs, silicate of soda of  $38^{\circ}$  B. 5 lbs.

Heat the stock of fat to  $180^{\circ}$  F. and dissolve in it the comminuted rosin over a moderate fire. Strain the rosined fat and weigh it. In the meanwhile bring the lye into the pan, allow it to boil through, and after withdrawing the fire, add the potash solution and the silicate of soda; then into the lye mixture, which should have a temperature of  $180^{\circ}$  F., succes-

sively crutch the rosined fat, which should show a temperature of about 158° F. The mass will finally be very thick, but vigorous crutching is not necessary. When the last portion of rosined fat has been brought into the lye remove the crutch, cover the pan for about two hours, then crutch again, and bring the soap into shallow frames covered with sheet-iron.

*Stock for rosined soap with 100 per cent. rosin and talc.*—Palm-kernel oil 50 lbs., rosin 50 lbs., talc 10 lbs., caustic soda lye of 37° B. 50 lbs.

This soap is made in a similar manner as the previous one. Heat the lye to about 176° F., stir in the talc and then crutch in the rosined fat at about 155° F. Allow the mass to stand in the covered pan for about one hour, then again crutch the mass, which will now be well combined and finally bring it into small frames.

*Stock for yellow rosined soap.*—Light bone fat 36 lbs., palm-kernel oil 14 lbs., crude palm oil 2 lbs., pale rosin 50 lbs., caustic soda lye of 36° B. 52 lbs., silicate of soda of 38° B. 2½ lbs., potash solution of 15° B. 2 lbs., brine of 15° B. 1 lb.

Melt the rosin in the stock of fat and strain the rosined fat through a sieve into a vessel. Then clean the pan, introduce the lye, silicate of soda, potash solution and brine, and bring to a boil; it is also advisable to melt some scrap in the hot mixture. When the lye mixture has cooled to about 158° F. and the rosined fat to about 149° F., gradually crutch the latter into the former. Then cover the pan for one hour, again crutch the mass, and finally bring it into the frame.

*Stock for red-yellow rosined soap.*—Ceylon cocoanut oil 150 lbs., crude palm oil 40 lbs., pale rosin 30 lbs., caustic soda lye of 30° B. 200 lbs., silicate of soda of 38° B. 75 lbs., water 40 lbs.

Heat in a quite roomy pan the lye, silicate of soda and water to boiling. In another pan heat the oil, melt the comminuted rosin in it and strain. Then, over a very moderate fire, gradually crutch the rosined fat into the boiling hot mixture of lye and silicate of soda. Great care should be observed in doing this, as the soap rises quickly in the pan on account of

combination taking place suddenly. When the soap subsides in the pan the saponification-process is finished. Samples tested upon a glass plate should be firm and show pressure. Should the soap lack firmness it is hardened by the addition of soda solution of 35° B. or soda crystals. During the cold season of the year, hardening has to be done with great care, too large an addition of soda solution readily causing efflorescence.

*Stock for pale rosined soap, so-called economy soap.*—Palm-kernel oil 317 pounds, tallow 42 pounds, palm oil 2 pounds, rosin 63 pounds, caustic soda lye of 34° B. 252 pounds, silicate of soda of 38° B. 84 pounds, brine of 24° B. 6 pounds.

This soap is prepared in the same way as the red-yellow soap. The lye, silicate of soda and brine are heated to boiling in one pan, and into this mixture is gradually crutched, at about 185° F., the rosined fat previously melted in another pan, and strained, the mass rising quite high in the pan. In case thorough combination should not have taken place when all the rosined fat has been introduced, the mass is allowed to remain for some time in the pan and is occasionally crutched till a well-combined soap has been obtained. The quite thick soap is finally brought into the frame and crutched till cold.

*Stock for brown rosined soap.*—Dark rosin 125 pounds, palm-kernel oil 90 pounds, wool fat 35 pounds, caustic soda lye of 39° B. 119 pounds, water about 15 pounds.

This soap is prepared in the same way as the two preceding varieties. It may also be made by heating the rosined fat as well as the lye to about 194° F. and crutching the latter into the former. When the soap, which at first rises in the pan, has subsided, crutch in 20 to 25 pounds of silicate of soda saturated with lye.

*Ivory soap.*—Most of the soaps now brought into commerce under this name are paste-soaps generally prepared by the cold process. A stock for such a soap consists of: Palm-kernel oil 100 pounds, caustic soda lye of 38° B. 50 pounds, solution of 25° B. 10 to 15 pounds, this solution consisting of boiling water

30 pounds, in which are dissolved potassium chloride 4 pounds, potash 4 pounds, and sugar 5 pounds. It is brought with cold water to 25° B., and only the clear portion of it is used.

The palm-kernel oil is heated to about 90° F., and then everything is stirred together. The proportion of lye given in the formula, as a rule, suffices, but should the soap unexpectedly turn out not sufficiently firm, 1 to 2 per cent. of lye of 38° B. may be added to the solution above described. A portion of the palm-kernel oil may also be replaced by tallow and the soap be more heavily filled even with silicate of soda, though it is not advisable.

## CHAPTER XIV.

### SOFT SOAPS.

By soft soap is understood a soap prepared chiefly from potash lye and oil, of a soft, salve-like consistency, and containing much more water chemically fixed than hard soap. It is not a simple alkaline sebate, but a solution of an alkaline sebate in a mixture of carbonated and caustic lyes. As neutral potassium oleate yields a viscid, gummy, and turbid product, it is necessary to add to it, in order to obtain the transparent soap of commerce, a solution of potassium hydroxide and carbonate, or to substitute for the latter one of allied salts. By depositing itself between the atoms of soap, the potassium carbonate, added in suitable proportion, breaks up the viscosity of the soap and forms it into a plastic, transparent mass, while it also possesses the property of combining water with the soap.

It is frequently stated that the glycerin contained in the oil and fat takes an active part in the process of saponification. Such, however, is not the case, it contributing at the utmost only to the transparency of the soap.

The principal fatty substances—train oil and hemp oil—which were formerly used in the manufacture of soft soap are but little employed at the present time, linseed oil, German sesame or cameline oil, rape oil, cottonseed oil, peanut oil, sesame oil, as well as oleic acid, tallow, palm oil, lard and horse fat being substituted for them. However, on account of its good qualities—it freezing only at about  $-4^{\circ}$  F.—linseed oil, besides cameline oil, is preferably used for winter soaps.

The success of the soap depends chiefly on the lyes, and it is necessary to prepare them with the greatest care, the prin-

cipal condition being to determine whether they contain correct proportions of caustic alkali and alkaline carbonate, or, as it is technically called, are "correct in lime."

For the saponification of 100 pounds of oil or fat about 36 pounds of 96 per cent. potash causticized by lime are generally required. If, however, the soft soap is to be filled, a considerable quantity of lye is necessary for saturation, so that 40 pounds of 96 per cent. potash must be allowed for 100 pounds of oil.

For boiling, lye of an average strength of 23° to 24° B. is generally used, 155 to 160 pounds of lye of 24° B. sufficing for 100 pounds of oil. Lyes taken directly from the tanks are not even in lime, the latter varying very much in lyes of 30°, 24° and 18° B.; the higher the degree of the lye the lower in lime it is. It being, however, absolutely necessary in boiling to keep within determined proportions of caustic alkali and alkaline carbonate, correspondingly weaker and stronger lyes are used; for instance, about 2 parts of lye of 20° B. and 1 part of lye of 30° B., by which a suitable average lye of about 24° B. is obtained.

The manner of preparing the various kinds of soft soap occurring in commerce being the same and carried on according to the same rules, they can only be divided into the following four varieties, according to the fatty substances used, and whether potash lye alone is employed or soda lye in connection with potash lye. 1. Smooth transparent soap. 2. Transparent soap with natural fig (natural fig soap). 3. Transparent soap with artificial fig (artificial fig soap). 4. Soap with a mother-of-pearl lustre (silver soap, elaidin soap). All these varieties demand a different treatment in the various seasons of the year, in order to present always the same appearance; it being of special importance to choose a different combination of fats for each season of the year, to pay special attention to the correct proportion of causticity of the lyes, the degree of moisture and the fit of the soaps, and to use the correct proportion of soda for smooth, artificial fig, and silver soaps.

1. *Smooth transparent soft soap*.—For this soap, which occurs in commerce as so-called crown soap of various colors (black, brown, green, etc.), linseed oil or cameline oil is generally used in winter, and sometimes train oil and hemp oil, these oils being nearly free from stearin and little inclined to congealing. In the warm season of the year, besides the mentioned oils, a considerable quantity of cottonseed oil, rape oil, peanut oil, sesame oil and olive oil is worked to impart to the soap greater consistency and durability. Smooth soft soaps prepared from linseed oil alone which, as pure potash soaps will stand any degree of cold, liquefy at a higher temperature, and to overcome this drawback a certain quantity of potash lye is replaced by soda lye. Various experiments in regard to the amount of soda lye allowable as an addition for soft soap have shown that a product of good quality can be obtained in summer by the use of 3 parts potash lye and 2 parts soda lye, while with the use of equal parts of potash and soda lyes, the soap becomes turbid and unsalable. At the present time one-fourth to one-third soda lye is generally used during the hot season of the year, which is, however, gradually reduced (to one-fifth, one-sixth, etc.) with the approach of colder weather, and entirely omitted when frost sets in, potash lye alone being then used. An allowance must, of course, be made for the soda naturally contained in the potash. It must also be borne in mind that soft soap, in the preparation of which at a low temperature too much soda lye is used, becomes short and friable, separates lye and acquires a turbid, bad appearance, and in freezing weather this defect may already happen with the use of one-fifth soda lye. Furthermore, while the clearness of the soap suffers the more, especially under the influence of cold, the greater the content of soda, a smaller yield is also obtained and the product is somewhat more expensive. By frequent weighing of different boilings it has been ascertained that a soap prepared with 6 parts of potash lye and 1 of soda lye yields at the utmost 230 pounds of soap from 100 pounds of linseed oil, and that the yield diminishes in a certain proportion with a higher percentage of soda lye.

Suppose a soft soap boiled from linseed oil and pure potash lye yields, when sufficiently evaporated, 235 pounds, and a pure soda soap only 190 pounds, the yield with the use of potash and soda lyes can be readily calculated.

1 part soda lye . . . . .	=	190 parts
6 parts potash lye @ 235 . . . . .	=	1410 "
<hr/>		
7 " in . . . . .	=	1600 " — 228½
Or 1 part soda lye . . . . .	=	190 parts
4 parts potash lye @ 235 . . . . .	=	940 "
<hr/>		
5 " in . . . . .	=	1130 " 226
Or 1 part soda lye . . . . .	=	190 parts
2 parts potash lye @ 235 . . . . .	=	470 "
<hr/>		
3 " in . . . . .	=	660 " . 220

Rosin (5 to 15 pounds to 100 pounds of oil) is frequently added to smooth, transparent soft soap, which not only renders it cheaper but also gives it a fine luster. The rosin is either at once added to the oil in the kettle and saponified with it, or is introduced with the required quantity of lye of 30° B. (about 92 pounds to 100 pounds of rosin) into the soap boiling up finished, and combined with it by crutching. The latter process is chiefly applied, as it gives paler soaps and a somewhat larger yield, though the soap has again to be fitted after adding the rosin. On the other hand, by boiling the rosin together with the oil, the soap becomes somewhat darker and the yield somewhat less, but the rosin is better incorporated with the soap, and a second fitting being not necessary the operation takes less time and is surer.

The use of rosin rendering the soap considerably softer, it is necessary to use besides potash lye a corresponding quantity of soda lye of 24° B., and the more of it the warmer the season of the year.

*Smooth soft soap from linseed-oil.*—In many parts of Northern Germany a smooth soft soap of a red-yellow, brown, or green

color is manufactured. In winter the soap is boiled with potash lye alone and quite strongly fitted with potash solution of 28° B., which not only protects it from freezing, but somewhat increases the yield. If rosin is to be worked in connection with the oil, it is generally saponified with soda lye. In the warmer season of the year oils and fats containing more stearin, such as cottonseed oil, peanut oil, oleic acid, horse fat, etc., are frequently worked in connection with linseed oil, whereby the soap acquires a better consistency and greater power to resist heat; some soda lye being also used as may be required.

Although in the hot summer months, and with the addition of a large quantity of rosin, up to one-third of soda lye is required, the proportion of it should be gradually reduced with the approach of cooler weather and entirely stopped in the cold season. Care must be exercised in the addition of such oils containing stearin, as above mentioned. In the hot summer months, 60 to 80 per cent. and eventually even more of cottonseed oil may be worked, but in that case pure potash lye only should be used.

To prepare the necessary lye for the saponification of 1000 pounds of linseed oil it is best, in order to be provided for all contingencies, to use 500 pounds of 96 per cent. potash and 216 pounds of lime. The lye obtained by the first lixiviation is generally set at 28° B. and that from the second at 20° B. Lye of an average strength of 23° B. is generally used, of which for the saponification of 100 pounds of linseed oil about 170 pounds (hence 100 pounds of 20° B. and 70 pounds of 28° B.) are required.

The boiling of the soap is carried on as follows: In the evening 1000 pounds of linseed oil and about 400 pounds of lye of 20° B. are brought into the kettle. The next morning the fire is started and the oil and lye are from time to time crutched through. When the mass is sufficiently hot an emulsion will be formed by crutching, the contents of the kettle begin to rise somewhat, and combination be established,

the latter being recognized by a sample not yielding lye and running from the paddle like a thick homogeneous ointment. When thorough and intimate combination has been established, 300 pounds more of lye of 20° B. are immediately added, and as soon as they have combined with the rest, 300 pounds more. The fire is now increased, and about 650 pounds of lye of 28° B. are introduced in three or four portions, care being had to allow each portion to combine before adding the next. When all the lye has been brought into the kettle, which will require about one hour, a dark but quite clear mass (paste) shows itself under a light cover of froth. Though a sample dropped upon glass is at first clear, it is still very thin and draws threads, and on cooling acquires a turbid appearance and becomes jelly-like. The froth upon the surface of the soap and the condition of the sample indicate that the soap still contains too large an excess of water. To remove this the fire is increased and the soap allowed to boil up high and to steam vigorously. The soap after boiling for some time appears clearer, thicker and shorter, the froth upon the surface disappears, and the soap begins to "talk," \* to boil into large plates and to break into roses.† When the soap is at this stage thrown with the paddle, it will show no water bubbles and run off in raised streaks, and break short from the paddle without drawing threads. When the soap, whilst boiling very gently, shows these signs and a sample upon a glass plate forms a small heap without many pearls of froth or air-bubbles, evaporation has been carried sufficiently far and fitting. *i. e.*, testing the soap as to whether the oil has been completely saponified by the alkali, is proceeded with. This is ascertained with greater ease with soft soaps than with soda soaps, as the latter become turbid on cooling.

Every drop of a pure oil soap shows exactly the same char-

\*The technical term for the noise produced by the steam developed on the bottom of the kettle in forcing its way through the thick soap.

†The thick soap on the place where the steam from below forces its way through resembles an expanding rose.

acteristics and behavior as the entire contents of the kettle, from above to below, would show only after a few days, and therefore the most accurate observations can be made on a clear sample. If the 1650 pounds of lye brought into the kettle have sufficed for the saponification of the 1000 pounds of oil, a sample dropped upon glass is clear and translucent, and, when held against a dark background, shows "flower" \* and a small ring of lye.† If the sample becomes turbid and whitish, and glides or is slippery on the glass, it is an indication of the soap containing too much lye. This is remedied by gradually adding oil to the boiling soap, until a sample shows the above characteristics of well-fitted soap. It is best to emulsify the oil with some weak lye before adding it, as in that state it combines better with the soap. If, on the other hand, the sample upon the glass is clear, but without "flower," it is a sure sign that lye is wanting.

After thoroughly examining and observing the samples, they are placed in the cellar, where the soap is, later on, to be stored, and in which an even temperature of from 50° to 59° F., should prevail. The samples of soap intended for the warm season of the year are generally placed near the ceiling of the cellar, and those for the colder season upon the floor. Soap for the warmer season must be mildly fitted to "small flower," and if the sample, after remaining half an hour in the cellar, is short, firm, and quite clear, it is correctly fitted. For winter soaps the fresh sample may show a somewhat stronger "flower," and the one cooled upon the stone floor of the cellar a considerable ring of lye; such soap better resists frost. If, however, the sample cooled in the cellar is clear around the edge and shows a turbid, milky point, the so-called "fat-gray" in the center, some lye is wanting. This is remedied by carefully adding to and boiling with the soap small portions of lye, until a sample dropped upon glass and cooled in the cellar is entirely clear.

\* The appearance of a striped skin or lye veins on the surface of the sample.

† A white ring the width of a hair surrounding the sample.

When the soap boils up finished, 5 to 15 per cent. of rosin with the required quantity of lye of 30° B. (about 92 pounds to 100 pounds of rosin), may be added, whilst constantly stirring and keeping the fire-door open. The soap must then be again fitted. Soda-lye is generally used for the rosin. In summer it is advisable not to exceed 10 per cent. of rosin, though in winter, and for soaps strongly filled with flour, somewhat more may be used.

The manufacture of smooth transparent soft soap from linseed oil having above been described, some disturbances which may happen in its preparation may be mentioned.

Although the previously mentioned proportion of lime will, as a rule, be found suitable, instances may arise where such is not the case. The "proportion of lime" is the chief trouble of the inexperienced soap-boiler, and causes him fresh anxiety with every boiling. It may happen that the lye is actually somewhat too caustic, or contains too much carbonate, and that this becomes perceptible in the soap-mass, so that a small correction is desirable. Frequently, however, the opinion that the proportion of lime is not correct is based upon a momentary deception, and the unsatisfactory appearance of the sample upon the glass is due to other causes, especially to insufficient evaporation and want of lye-strength, which misleads the inexperienced soap-boiler, and makes him think the lye is "too high in lime." He will, however, acquire the necessary experience and accuracy by close observation and more practice.

That the soap is too high in lime is recognized by its boiling sluggishly farther down in the kettle, rising only with a very strong fire, and running from the paddle in broad, viscid streaks, and a sample dropped upon glass lying high, and being turbid and gummy after cooling. Normal boiling of the soap is restored by successively adding potash-solution of 26° B., until the samples dropped upon glass are short, firm, clear, and pliant. Should, on the other hand, the soap be "to low in lime," it boils up high, and rises over, so that it

can be scarcely curbed, has a thinly-fluid, aqueous appearance, and a sample dropped upon glass runs. This is best remedied by an addition of pure caustic potash-lye, or weak caustic tank-lye, or, when the soap does not contain too much soda, of caustic soda-lye of 24° B., and in summer also by oleic acid. It is, however, always advisable not to be too hasty in making corrections in the proportion of lime, but only after being convinced of the necessity by quite a strong evaporation of the soap.

Thickening of the soap may also be briefly referred to. Such thickening especially happens after combination has taken place, and is due to the weaker more caustic lye charged together with the oil combining with the latter with great ease and extreme rapidity, and the mass is then lacking in phlegm. Hence when combination sets in and small steam-bubbles making a noise evolve from the bottom of the kettle, more lye, for which the mass shows great avidity, has to be quickly added as otherwise the entire contents of the kettle will lump together. However, if the soap has once become thick, it liquefies with difficulty notwithstanding the immediate addition of a larger quantity of lye and some lumps will not dissolve till an excess of lye, *i. e.*, more than is required for the saponification of the stock of fat in the kettle, has been added to the soap whilst vigorously boiling. Somewhat excessive boiling does no harm, the soap becoming thereby slightly paler, while, on the other hand, the appearance of a rather weak soap suffers more from the heat of the kettle when boiled for a longer time. The excess of lye-strength in the soap can near the end of the operation be readily overcome by the addition of some oil.

Great attention must be paid to the sufficient evaporation of the soap, for a product containing too much water remains soft, and on storing becomes turbid and thin and draws threads. The soap must be evaporated until no more froth is visible upon the surface, and samples dropped upon glass show but few pearls of froth and are sufficiently clear and firm;

small traces of froth on the walls of the kettle are of no importance.

Fitting the soap is also of importance for its future behavior. Soap too slightly fitted becomes turbid, soft, and liquid in the barrels, while when too strongly fitted it becomes short, friable, and eventually separates lye on the surface. In fitting the soap the season of the year at which it is to be sold must be especially taken into consideration. In the cold season of the year it must be strongly fitted to prevent it from freezing; in summer, however, the same soap cannot be too mildly fitted, as it would liquefy at a temperature of over 77° F. if it did not contain sufficient soda or oils containing stearin. In cold weather the final fitting is carried out as much as possible with potash solution of 28° B. A sample dropped upon glass may show a strong "flower" and ring of lye, but, of course, fitting must not be carried too far. In summer the soap is only sufficiently fitted to insure its keeping; a sample should show only slight "flower," and after cooling remain perfectly clear.

The proportion of soda is of importance as regards the durability of the soap, as well as the yield and the profit to be derived from it. While formerly the consistency of the soap was regulated by the use of softer or more solid fats and boiling with potash lye alone, at the present time the same oil is frequently used both in summer and winter, but the proportion of the soda to potash is changed. Both the solidity and the yield depend on the use of the soda, however, in an inverse ratio; the more soda lye is used the more firm the soap becomes, but the smaller the yield. In midsummer about 50 pounds of the soda lye of 24° B. are taken for every 100 pounds of oil, whereas in winter at the utmost 25 pounds. In continued severe weather, and when working silicate of soda and oils or fats containing stearin in connection with the oil, soda lye is either entirely omitted or the proportion very much reduced.

When rosin is added to the soap, as is generally done, 80

pounds of soda-lye of 24° B. are allowed for every 100 pounds of it; because the soap would become too soft with the use of potash lye for the saponification of the rosin.

Great care has to be observed in the use of soda lye for soft soaps in winter, because when too much of it is taken it will be detrimental to the appearance of the soap; it becomes too short, does not hold the lye, but lets it go, presents a crummy and yellow appearance and becomes unsalable.

In conclusion it may be said that the process of preparing all kinds of soft soap, no matter what name may be applied to them, is exactly the same as that described for the product from linseed oil, and that in order to obtain a firm and stable article, due consideration has to be given to the proportion of lime, content of moisture, fitting and prevailing temperature.

*Artificial fig soap.*—In commerce this soap occurs of a yellow, green or brown color. It differs very much as to quality, some products being transparent and unfilled, while others contain varying quantities of filling. For its preparation serve linseed, caneline, cottonseed, rape, sesame, peanut and sulphur-olive oils, and sometimes also oleic acid, saponification being effected with potash lye and eventually with 20 to 25 percent. soda lye. As a rule, 10 to 15 per cent. of dark or pale rosin is used in connection with the oils. The artificial figging used for this soap is usually prepared from chalc or thoroughly burnt lime. Of the chalc figging various sizes are found in commerce. It is hard, dissolves with difficulty, and sinks, when using the soap, to the bottom of the wash-tub. Lime figging, on the other hand, shows none of these disagreeable properties, but as it cannot be had in commerce because the lime when exposed to the air or moisture readily slakes, the soap-boiler has to prepare it himself. For this purpose well-burnt lime is pounded, passed through a medium-meshed wire sieve, and then freed from lime dust by means of a hair-sieve. The small irregular pieces remaining in the hair-sieve form the figging, and are preserved for future use in a large well-stoppered carboy. Later on 3½ to 14 ounces of them are

are added to about 200 pounds of the soap when somewhat cooled. The lime soon slaking in the warm, liquid soap expands and forms figging similar to the natural one.

*Yellow artificial fig soap.*—This is a golden-yellow, transparent soap, which is chiefly prepared from refined cottonseed oil and bleached linseed oil and potash lye, some crude palm oil being generally used for coloring.

The soap is prepared in a similar manner as given for smooth, transparent soft soap. Combine 1000 pounds of oil with lye of 15° to 16° B. over a moderate fire. When combination is established, add quickly more lye of 20° to 24° B. in portions of 200 to 300 pounds, allowing one portion to combine before adding the next, until a clear paste is formed. The fire being increased, allow the mass to boil high, whereby the froth will gradually disappear and the paste improve in clearness and consistency. By continued boiling the soap will soon be freed from superfluous water and froth, boil in large plates, audibly “break into roses,” and, when thrown with the paddle, break off short and have a woolly appearance. At this stage the palm oil required for coloring is added to the white soap while gently boiling, and the latter then fitted to “flower.” With a correct fit, samples upon the glass must show a “perceptible flower,” and after cooling be clear, short, and firm.

If the samples are transparent on the surface but turbid in the center, the soap is too weak, and lye has to be carefully added until it shows sufficient “flower” and remains clear on cooling. If, however, the sample runs over quickly, is turbid after cooling, and short and slippery upon the glass, the soap is too sharp, and oil mixed with weak lye has to be added until a sample remains entirely clear after cooling.

Crutch into the finished soap, cooled to about 167° F., 5 to 6 pounds of artificial figging and pour into dry barrels.

*Green artificial fig soap.*—Formerly this soap was prepared from hempseed oil, but as the supply of this oil decreased, linseed oil was substituted and the resulting yellow soap colored

green chiefly with indigo prepared in the following manner : For a stock of 1000 pounds of oil from 8 to 10 ounces of a good grade of indigo were rubbed fine and boiled with lye of  $23^{\circ}$  to  $24^{\circ}$  B. until the whole had acquired a soapy consistency. A drop of this boiled indigo added to a glassful of rain or river water had to produce a green coloration ; if the water acquired, however, a more bluish-green color, some more lye was added to the indigo and the whole boiled for some time longer. The indigo thus prepared was diluted with some lye and added to the finished soap, giving it a beautiful green color.

In modern times the soap is generally colored with ultramarine blue mixed with water or lye, 2000 pounds of oil requiring about 1 to 2 pounds of ultramarine for prime green soap.

Besides indigo or ultramarine-blue, chlorophyll, as well as the very intense hot water-soluble greens F and M are also used for coloring soft soaps green. A permanent fine green color may also be imparted by boiling together with the stock about 10 per cent. of sulphur olive oil. For winter soaps this oil, which is very rich in stearin, must previously be prepared as follows : Heat the oil, bring it into a barrel and cover the latter. In about eight days the solid portions of stearin and palmitin settle, and the liquid supernatant oil is used for winter soaps and the solid portion for summer soaps.

In winter linseed and cameline-oils alone are used for the preparation of green soap ; in summer a part of these oils is, however, replaced by cotton-seed oil, rape-oil, and sesame oil, as well as by horse-fat and train-oil. In winter the soap is generally boiled with potash lye alone, but in summer 20 to 30 per cent. of soda lye is used to give the soap a better consistency. At the approach of the colder season of the year the addition of soda lye must be gradually diminished, as otherwise the soap might become turbid and slippery ; for heavily filled soaps little soda lye is used.

The lyes are prepared in the same manner as for other soft soaps. The lime is slaked in hot tank lye of  $7^{\circ}$  to  $8^{\circ}$  B. and

the potash dissolved in the resulting milk of lime. Since 100 pounds of 96 per cent. potash saponify about 270 pounds of oil, about 400 pounds of 96 per cent. potash are required for 1000 pounds of oil, and about 170 pounds for causticizing the potassium carbonate. However, in order to have some lye in reserve in case of emergency, it is advisable to set 500 pounds of 96 per cent. potash and 215 pounds of lime. The soap is boiled with an average lye of 23° to 24° B., about 160 to 165 pounds being required for the saponification of 100 pounds of oil. With the use of 50° caustic potash lye about 18 lbs. of refined 96 per cent. potash are required for every 100 lbs. of it to reduce causticity. If the soap is to be filled the addition of potash is suitably reduced—about  $\frac{1}{4}$  to  $\frac{1}{3}$ .

The soap is prepared as follows: Bring 1000 lbs. of oil together with 400 lbs. of lye of 18° to 20° B. into the kettle and allow to stand over night, a kind of emulsion being thereby formed. The next morning combine the oil and lye by moderate boiling, and when combination is established add more lye. By careful firing and adding lye at the proper time thickening of the mass is prevented. After gradually adding more lye of 27° to 28° B., and increasing the fire, paste is formed, which will become clearer and of a better consistency by the addition of more lye and continued boiling. The required lye having been gradually added, the fire is increased and the soap thoroughly boiled to remove the superfluous water. With a kettle wide on the top, which offers to the clear boiling soap a large surface for evaporation, the work proceeds rapidly, the froth will soon disappear, and the soap boil in plates and commence to "talk." The soap is finally fitted to a "good flower," so that a cooled sample is clear and firm and does not become turbid after standing for some time.

Soap sufficiently evaporated and boiled to a finish should not show froth on the surface, should boil in large plates, audibly break into roses, when curbed show no bubbles, and run from the paddle held in a perpendicular position in raised streaks breaking off short. A sample upon glass should have

flower, and after cooling and lying for some time be sufficiently short and firm. If the soap is not to be further augmented, lyes containing more carbonic acid are used for them, especially in winter, the result being not only a better appearance of the soap but it also becomes more transparent and is better protected against frost. If, on the other hand, the soap is to be augmented by the addition of rosin or silicates more caustic lye and more vigorous evaporation are required.

If rosin is to be added to the soap, about 100 pounds of comminuted pale rosin, together with 92 pounds of lye of 30° B., are added to the finished soap and the whole boiled through. The soap is then once more carefully fitted. The addition of rosin renders the soap soft, and it is necessary to use one-fifth to one-third of soda lye of 24° B., according to the season of the year and the amount of rosin.

The finished soap is colored in the manner previously described and allowed to rest for some time. When cooled to about 155° to 167° F. the lime-flogging (1 pound to 1000 pounds of oil) is crutched in, the soap perfumed with some oil of mirbane, and then poured into dry barrels.

A second quality of green soap with a yield of 300 to 325 lbs. for every 100 lbs. of oil is made in the same manner, but 10 to 15 per cent. pale American rosin and flour filling are used, and for 1000 lbs. of oil-stock the basis soap is first colored with 2 ozs. of soap-yellow, and 1 to 1½ lbs. of ultramarine blue are then stirred in together with the potato-flour. Double the quantity of lime-flogging has to be crutched into such filled soap.

*Brown artificial fig soap* is also found in commerce. It is prepared by boiling dark rosin or some wool-fat together with the oil, or coloring the finished soap with sugar color.

*Natural fig soap.* This is without doubt the best of all kinds of soft soap. Its manufacture is, however, connected with difficulties, and the knowledge required for the preparation of a uniform and faultless product can only be acquired by considerable experience and close observation in boiling.

In the manufacture of this soap both hard fats containing stearin and softer fats and oils are used, the first yielding the "figging" and the latter the basis-soap. Good materials only should be used, the tallow especially requiring to be as fresh as possible and free from dirt and acidity. Old tallow which has undergone a partial decomposition should be purified and prepared before use. This is done by melting it and then heating to 189.5° F., and adding, with constant stirring, 1½ pounds of soda lye of 38° B., previously mixed with about 7 ounces of common salt for every 100 pounds of tallow. When the tallow has somewhat subsided, add boiling water (6 pounds to 100 pounds of tallow), cover the kettle, and after standing a few hours take the tallow off. Tallow prepared in this manner is well adapted for the manufacture of natural fig soap. If the soapmaker is, however, forced to use large quantities of old tallow without being able to purify it before use, it is advisable to work somewhat more carbonated lyes, any excess of carbonic acid being expelled in boiling the soap by the free oleic acid of the tallow. Horse-fat, lard and bone-fat, which are frequently used, especially in summer, to make the soap more compact and able to resist the heat, must also be purified before use.

Other materials used are clear linseed oil, or cameline oil, and in summer one part of thoroughly refined cotton-seed oil. Saponified oleic acid, which otherwise is an excellent material in connection with other oils, especially for milling soaps, is apt to make the soap somewhat dark, and must therefore be omitted where a light product is demanded.

The potash used in the manufacture of this soap should not contain more than two to three per cent. of soda, as otherwise the figging will be small and feathery, and the soap may even turn out entirely smooth. It is therefore advisable to procure from a reputable firm 96 to 98 per cent. potash guaranteed to contain only one-half per cent. of soda.

For the preparation of the lyes thoroughly burnt lime free from sand and grit is required. Slake the lime with hot tank-

lye of 6° to 8° B. and dissolve in this the required quantity of 96 per cent. potash. With good lime, and tolerably good water, 44 to 45 pounds of lime suffice in summer for 100 pounds of 96 per cent. potash, and 40 to 42 pounds in winter.

A solution of 400 pounds of 96 per cent. potash is sufficient for a stock of 1000 pounds of fats. From 100 pounds of 96 per cent. potash 500 pounds of lye of 24° B. are on an average obtained, 155 to 160 pounds of which saponify 100 pounds of fat.

The portion of the lye first obtained by dissolving the potash is drawn off and set at 27° to 28° B., and is used for boiling and filling, while the second portion is set at 17° to 18° B., and employed as combining lye. Lye of less strength than 17° B. is as a rule not used, because too much moisture would have to be evaporated and, besides lyes of 12° to 15° B. contain a considerable quantity of foreign salts in solution which in boiling act the same as carbonates, and frequently exert even a stronger influence. If nevertheless, the weaker tank lyes are to be utilized it is advisable to evaporate them to about 25° B., the salts precipitating from such more concentrated lye.

When the lyes have been prepared a fixed quantity of stock is made ready for boiling. The composition of the stock varies very much and depends on whether the soap is to be boiled for the colder or warmer season of the year, whether a larger or smaller figging is desired, and whether the soap is to be of a more light or dark yellow color.

As regards the figging it must be borne in mind that its size in properly boiled soaps, *i. e.* in soaps in which the content of water, the proportion of carbonic acid and the fitting are correct, depends entirely on the combination of the stock of fat. The more fat rich in stearin is used in proportion to oil, the more figging is obtained in the soap, though it will also be accordingly denser and smaller, because space for its greater development is lacking.

Below some stocks suitable for various conditions are given:

1. *For pale yellow soap with rice-like figging.* In summer: Tallow 35 per cent., crude palm oil 3, pale horse fat or hog fat 16, cottonseed oil 16, pale linseed oil 30.

In winter: Tallow 35 per cent., crude palm oil 2, cottonseed oil 15, pale linseed oil 48.

2. *For dark yellow soaps with rice-like figging.* In summer: Tallow 36 per cent., crude palm oil 2, horse fat 20, linseed oil 42.

In winter: Tallow 35 per cent., crude palm oil 3, linseed oil 62.

3. *For pale soaps with small rye-like figging.* In summer: Tallow 48 per cent., crude palm oil 2, cottonseed oil 15, pale linseed oil 35.

In winter: Tallow 40 per cent., crude palm oil 1, horse fat or hog fat 10, pale linseed oil 50.

4. *For dark soap with small rye-like figging.* In summer: Tallow 45 per cent., crude palm-oil 5, linseed-oil or cameline-oil 50.

In winter: Tallow 40 per cent., crude palm-oil 5, linseed-oil or cameline-oil 55.

5. *For soap with a beautiful medium figging.* In summer: Tallow 40 per cent., cotton-seed oil 25, pale linseed-oil or cameline-oil 35, crude palm-oil for coloring 1 to 2.

In winter: Tallow 40 per cent., linseed-oil or cameline-oil 60, crude palm-oil for coloring 1.

6. *For soaps for milling purposes.* In summer: Tallow 34 per cent., palm-oil 6, oleic acid 40, cotton-seed oil 20.

In winter: Tallow 30 per cent., palm-oil 5, horse fat or oleic acid 30, peanut oil or bleached linseed-oil 35.

Before entering upon the description of the process of boiling natural fig soap, the conditions that have to be closely observed in order to obtain a good product may be briefly referred to. Special attention must be paid to the proportion of lime required, the necessary content of moisture and normal fitting.

As mentioned in speaking of smooth transparent soaps, a

salable soft soap cannot be prepared from pure caustic potash lye, as the result would be a gummy, viscid mass. To prevent this viscosity, and to give the product the required pliancy and salve-like consistency, a sufficient quantity of potassium carbonate in solution must be present, which, by depositing itself between the atoms of soap, loosens the combination. This holds also good, but in a still greater degree, as regards natural fig soap. In the preparation of this soap,  $\frac{1}{2}$  to  $\frac{1}{3}$  tallow is used, which always contains more or less free fatty acid, and for this reason such soap must contain a proportionally larger quantity of potassium salts in solution than smooth transparent soap, and the more so as on this depends the ability of the potassium stearate (figging) to move and develop, or, in other words, the possibility of crystallization and the formation of figging. Hence the more solid fat rich in stearin (tallow, etc.) in proportion to soft fat (oil, etc.) is used, the more carbonate the lyes employed for saponification must contain.

In the foregoing too great causticity in the soap has been referred to, and now the opposite case will have to be considered. As previously mentioned, the potassium salts, by depositing themselves between the atoms of the soap, somewhat loosen the combination and break up the viscosity. The more potassium salts are introduced into the soap-mass, the looser and softer the latter naturally becomes, and with a further increase of potassium salts, combination is effectually destroyed, the potassium salts are eliminated and the soap becomes finally salted out. Hence, in order not to destroy the consistency of the soap-mass, the potassium salts must always be present only in a fixed proportion to the caustic potash.

The tank lyes prepared by the soapboiler himself contain the more carbonic acid (hence are lower in lime) the more high-graded they are and are the more caustic (hence higher in lime) the weaker they are. Pure caustic lyes, as previously mentioned, are not serviceable for the manufacture of soft soap, and they must always contain a definite portion of car-

bonates in solution. Tank-lyes possess these requisites and in order to establish the required proportion between caustic potash and carbonate of potash, and to obtain a good product, all that is necessary is the correct use of suitable quantities of a lyes more caustic and of one containing more carbonic acid.

For the manufacture of natural fig soap, as previously mentioned, lye of 16° to 18° B., being more caustic, is used as combining lye, and one of 28° to 30° B., containing more carbonic acid, as boiling lye, and in order to be quite right as regards the proportion of lime, enough of both kinds is, as a rule, taken to make an average lye of 23° to 24° B.

The operation of boiling with ease and rapidity with the use of an average lye of 23° to 24° B. is as follows: After once using a certain lye-pot for a boiling of soap, it will be known how many potfuls of lye of 23° to 24° B. are required for the saponification of say, 220 pounds of fat. By noting the number of pots, the quantity of lye required for the next boiling can be quickly brought into the kettle, and evaporation of the soap proceeded with. Suppose a stock of 2200 pounds of fat is brought into the kettle, and 8 pots of lye of 24° B. are required for the saponification of 220 pounds of fat, then 34 potfuls of lye of 18° B. are added to the fat in the kettle, and combination is promoted by crutting over a moderate fire. A thorough combination between fat and lye being established, 42 potfuls of lye of 28° B. are gradually added in portions of 10 to 12 potfuls with vigorous boiling, whereby paste will be formed. After thoroughly boiling the soap, about 5 potfuls of lye, which are still required, are gradually added, and, a sample being from time to time taken upon the glass, the sufficiently evaporated soap is finally fitted to a "slight flower."

The average lye is calculated in the following manner, according to the number of potfuls of lye of 18° and 28° B. which were required for the complete saponification of the fat:—

$$\begin{array}{r}
 34 \times 18^\circ \quad . \quad . \quad . \quad . \quad = 612 \\
 46 \times 28^\circ \quad . \quad . \quad . \quad . \quad = 1308 \\
 \hline
 80 \text{ divided into} \quad . \quad . \quad . \quad . \quad \overline{1920 = 24}
 \end{array}$$

Hence, by dividing 80 (the number of potfuls of lye used) into 1920, the figure 24 is obtained, which, in this instance indicates the strength of the average lye.

The process of boiling these soaps and the contingencies which may arise during the operation will now be discussed.

When with the use of a moderate fire combination has been established, lye of 28° B. must be at once added as otherwise the soap might readily thicken, and this addition of lye is continued till paste is gradually formed. The soap is now allowed to boil high for the evaporation of superfluous water. After adding almost the entire quantity of lye still required, and boiling for some time, the soap will become thicker, clearer, and shorter. The froth upon the surface of the soap, which is an indication of excess of water, will gradually disappear, the soap commence to boil in larger plates and audibly break into roses.

While the soap is boiling notice has to be taken as to whether it stands "correct in lime." Soap "too high in lime," even if thoroughly evaporated, is viscid and gummy after standing a few hours, and remains turbid and figs badly; soap "too low in lime" figs quickly, but shows a small and more round figging, and the transparent basis-soap, lacking the required cohesion and consistency, becomes readily soft and syrupy.

Soap "too high in lime" boils sluggishly lower down in the kettle, and rises only with a very strong fire. Further, the soap runs from the paddle in broad viscid streaks, and a sample dropped upon the glass will pile up high, congeal quickly, and be gummy and viscid. On the other hand, if the soap is "too low" in lime, it boils up high, rises over, is thinly-fluid, and samples dropped upon the glass spread out. Soap "correct in lime" boils easily and nicely, runs readily from the paddle in raised streaks, lies for some time upon the glass, so that the rings formed can be observed until cooling; the cooled sample breaks short like grained soap. Hence, if natural fig soap does not boil normally, solution of potash of

28° B. or caustic potash lye of 23° B. must be added according to requirement until it shows the requisite properties.

When the soap audibly "breaks into roses" and shows a correct proportion of lime, attention must be directed towards evaporation. A soap too strongly evaporated, and consequently deprived of the necessary content of water, figs very slowly and with difficulty, and sometimes not at all, on account of being too solid. If, on the other hand, the soap has sufficient moisture, the separation of the potassium stearate (figging) takes place with corresponding rapidity and regularity. In evaporating, the soap-boiler must principally be guided by the composition of the stock of fats; the more solid fat, which fixes moisture, has been used the less the soap is to be evaporated. It is sufficiently evaporated when it boils in plates, audibly "breaks into roses," runs from the paddle in raised streaks, and slips off from it short and without spinning (drawing threads). It should also adhere in small drops to the finger dipped into a sample upon the glass.

The soap being sufficiently evaporated, it is adjusted to "flower." Samples upon the glass must show a "perceptible flower," be clear and firm after cooling, and after standing for some time appear no longer bright, but as if breathed upon. Special care must be had in fitting, for if the soap is too sharp the more delicate crystals of palmitic acid contained in the fats, which are actually intended to make the soap more compact, would also incline towards the formation of figging, so that the soap would contain too much and wild figging, and, further, become slippery and separate lye. On the other hand, soap too slightly fitted, though forming good figging, readily becomes soft and liquid. The soap is too sharp when a sample upon the glass runs over immediately and clarifies with difficulty or not at all; it is too weak when a sample upon the glass is not entirely clear after cooling, but shows a turbid point—the so-called "fat gray"—in the center. Any necessary correction is made by adding fat, or lye of 24° B., as may be required. A sample upon the glass must show just sufficient "flower" to insure the durability of the soap.

If the soap is to be colored, the necessary quantity of crude palm oil, together with the required quantity of lye, is added towards the end of boiling.

The finished soap is allowed to rest for some time, slightly perfumed with oil of mirbane, and then poured, at a temperature of about 155° F., into the barrels. The yield of unfilled natural fig soap amounts to from 235 to 240 pounds from 100 pounds of fat.

The barrels containing the soap are then brought into the cellar, the temperature of which must be kept at from 54.5° to 66° F. in order to promote the formation of figging. At below 54.5° F. the crystals in the soap would congeal too quickly, and at over 66° F. remain in solution. The figging, which is a crystalline separation of potassium stearate and palmitate, forms in three to eight weeks, according to the proportion of tallow in the soap.

It may finally be remarked that in boiling natural fig soap the temperature of the various seasons of the year must be taken into consideration, as, for instance, soap boiled in summer sometimes becomes turbid and wet by October. There is no one method of boiling natural fig soap that is suitable for all cases, it being necessary to take into consideration the composition of the stock of fat and the proportion of lime required for the appropriate season of the year.

*Alabaster natural fig soap.* Under this name a soap is brought into commerce which shows snow-white figging in a whitish-yellow, very transparent basis-soap. It is very pure and of fine quality, as by filling it would suffer in appearance and formation of figging.

The stock for this soap consists best of 1 part prime mutton tallow and 2 parts prime, pale cotton-seed oil. It is advisable to use caustic-potash lye of 50° B., and for reducing the causticity about 20 per cent. of refined 98 per cent. potash free from soda is required. The boiling lye for the soap is mostly set at 25° B., and 150 lbs. of it are necessary for 100 lbs. of stock of fat. The combining lye is readily obtained by adding the necessary quantity of water to the lye of 25° B.

The soap is prepared like every other kind of natural fig soap. For instance, 800 pounds of best quality of mutton tallow and about 1600 pounds of prime pale cottonseed oil are brought into the kettle together with about 800 pounds of lye of 18° to 19° B. and combined with the use of a moderate fire. When combination has been established, more lye of 25° B. in portions of 400 to 600 pounds is quickly added, boiling being steadily kept up, till a clear soap paste is in the kettle. The fire is then increased and the mass allowed to boil high; the froth will gradually disappear and the paste gain in clearness and consistency. The soap freed from excess of water will soon be free from froth, boil in large plates, audibly break into roses and, when thrown from the paddle, fall down short and show a woolly appearance. When pushing the paddle through the soap, the latter will rustle and, when taken upon the paddle, run from it in raised streaks and break off short without drawing threads. At this stage fitting is proceeded with; this should, however, be only light, because soaps from tallow and cottonseed oil will not stand vigorous fitting. Samples upon the glass should have a light flower, be clear when heaped up, and when cold be sufficiently firm.

The finished soap is immediately brought into barrels standing in a room in which a constant temperature of 64° to 68° F. prevails. Besides sufficient causticity, this temperature is particularly necessary for alabaster soap so that it will always remain clear and transparent and the figging process take place slowly and regularly.

*Natural fig soap with stearin.*—In the same manner as tallow, pure stearin may also be used in the preparation of natural fig soap, the figging obtained being especially firm and stable.

The process of manufacture is as follows: 1200 lbs. of linseed or cameline oil—in summer also  $\frac{1}{2}$  cotton-seed oil and eventually some horse fat—are brought into the kettle together with about 500 lbs. of lye of 20° B., and combined over a moderate fire. Combination being established more lye is quickly added to prevent thickening of the mass. The fire

being increased, more lye of 20° B. is introduced in portions until paste is formed, which is thoroughly boiled, with the successive addition of the quantity of lye of 28° B. still required, until the superfluous water is evaporated. When no more froth is noticed upon the surface, and the soap boils in plates and audibly "breaks into roses," and when thrown shows no more water-bubbles, but runs in raised streaks from the paddle and breaks off short, fitting is proceeded with. With a correct fit the samples upon the glass must be clear, firm, and short, show a good "flower," and remain clear after cooling. To the soap when boiling up finished, add 100 pounds of stearin, together with about 125 pounds of potash solution of 40° B., and allow the whole to combine. A sample upon the glass must again show a "good flower" and remain clear after cooling.

The finished soap, when cooled to about 156° F., is poured into barrels and the latter brought into the cellar, where, with a suitable temperature, the figging will soon form.

*Soft soap with a mother-of-pearl luster.*—This soap, also known as elædin soap, silver soap, etc., is much liked for household purposes, as well as in the textile industries. In its preparation various stocks of fats are used, consisting of tallow, horse-fat, lard, bone-fat, oleic acid, palm-oil, linseed-oil, cameline-oil, peanut-oil, and cotton-seed oil, the latter being much used when it can be obtained at a sufficiently low price. Palm-oil is also worked to a considerable extent, it being used in the crude state for giving the soap a yellowish color, and in the bleached state for imparting to it an agreeable odor.

The lyes required for this kind of soap are prepared in the same way as given for other soaps. The lime, about 42 lbs. for every 100 lbs. of .96 to .98 per cent. potash is slaked with hot tank-lye of 7° to 8° B., and the potash dissolved in it. For boiling, potash lye of 20° to 25° B. is generally used in connection with one-third to one-sixth of soda lye of 25° B. according to the season of the year and the amount of filling. In working large quantities of solid fats a suitable reduction

of the proportion of soda is required. The radiated silvery appearance of the soap is produced by the soda lye, the sodium salts combining with the particles of stearin and palmitin in the fats to white silvery streaks. The soap acquires the silvery appearance only after complete cooling and storing for 8 to 14 days.

The stock of fat for this kind of soap has to be chosen so as to suit the prevailing temperature and season of the year. Below a few suitable stocks are given.

*For white soap.* I. *In summer.* Prime pale cotton-seed oil 75 lbs., tallow 25 lbs.—*In winter.* Prime pale cotton-seed oil 80 lbs., pale hog-fat 20 lbs.

II. *In Summer:* Prime pale American cotton-seed oil 65 lbs., tallow 30 lbs., palm-kernel oil 5 lbs.—*In winter:* Prime pale cotton-seed oil 75 lbs., hog-fat 20 lbs., bleached palm oil 5 lbs.

III. *In Summer:* Prime pale cotton-seed oil 60 lbs., pale hog-fat 35 lbs., bleached palm oil 5 lbs.—*In winter:* Prime pale cotton-seed oil 60 lbs., peanut oil 20 lbs., pale hog-fat 20 lbs.

IV. *In summer.* Pale cotton-seed oil 60 lbs., tallow 20 lbs., hog-fat 20 lbs.—*In winter:* Pale cotton-seed oil 75 lbs., tallow 15 lbs., bleached palm oil 10.

V. *In Summer:* Pale cotton-seed 50 lbs., pale horse-fat\* or hog-fat 50 lbs.—*In winter:* Pale cotton-seed oil 90 pounds, tallow 10 pounds.

*For yellow soap.*—I. *In summer:* Cottonseed oil 60 lbs., tallow 35 lbs., crude palm oil 5 lbs. *In winter:* Cottonseed oil 75 lbs., tallow 20 lbs., crude palm oil 10 lbs.

II. *In summer:* Cottonseed oil 60 lbs., horse fat 35 lbs., palm oil 5 lbs. *In winter:* Cottonseed oil 80 lbs., palm-kernel oil 15 lbs., palm oil 5 lbs.

*For a milling soap of good quality.*—*In summer:* Cottonseed

\* By horse fat is understood a mixture of horse-fat tallow, and lard as furnished by establishments where dead animals are boiled.

oil 75 lbs., tallow 15 lbs., bleached palm oil 8 lbs., palm-kernel oil 2 lbs. *In winter:* Cottonseed oil 60 lbs., horse fat or hog fat 30 lbs., tallow 10 lbs.

In preparing these soaps the proportion of lime first deserves attention. To produce a beautiful, delicate and white soap more strongly carbonated lyes are required for boiling, and as a soap "too high" in lime would turn out too solid and dark, the proportion of carbonate to caustic potash in the lyes should be about as 1 : 2. As the soapboiler does not like to change the proportion of lye to which he is accustomed, he can boil with his ordinary tank lye and bring the soap to the required proportion of lime by the addition of potash solution of 24° B. If the soap has the right proportion of lime or causticity, and been regularly boiled and fitted, samples the size of a dollar will heap lightly upon the glass and after standing for about five minutes remain still somewhat liquid in the center.

The addition of soda lye varies according to the composition of the stock of fat and the season of the year, less being required the more tallow is used, while for soaps chiefly prepared from cottonseed oil, lard and horse fat, as much as 33 pounds of soda lye of 25° B. may be worked for every 100 pounds of fat. With a stock consisting of tallow 90 parts and palm-kernel oil about 10 parts, no soda lye at all is, for instance, used, but for the production of a smooth, delicate product, the soap must be kept somewhat more carbonated.

Special attention must also be paid to fitting the soap. Samples upon the glass should show only perceptible flower, be sufficiently clear, firm and short, become white only after some time, and not be readily pushed from the glass but adhere firmly to it. Soap too strongly fitted would become short later on and separate lye; on the other hand, fitting must not be too weak as the soap, when stored, would become soft and "long." Any corrections required in fitting can readily be made by small additions of fat or lye.

The soap is as a rule, only evaporated till it breaks into roses. It is then allowed to stand quietly over night in the

covered kettle. The next morning any froth which may have collected on the surface is removed and a slight correction, if necessary, made by the addition of potash lye or potash solution. The yield amounts to about 235 to 240 lbs. of soap from every 100 lbs. of stock of fat. However, in most cases it might be better to finish evaporation only when the soap boils free from froth and uniformly breaks into roses over the entire surface. On the other hand, evaporation continued too long is not advisable as it would only make the soap gray and dark.

Sometimes 5 to 10 per cent. of pale rosin is added to soft soaps for domestic use. The rosin, to be sure, does not impair the quality of the soap, but as soaps with an addition of it fix less water, no material advantage is by this practice derived.

If in the preparation of this soap potash lye of 50° B. is to be used, for every 100 lbs. of it 25 lbs. of refined 96 to 98 per cent. potash are required for reduction of causticity, and 18 to 20 lbs. for highly filled soaps. The resulting reduced lye is by the addition of water brought to 25° B., and of this 150 lbs. are required for the saponification of the stock of fat. In addition there will, as a rule, be worked for every 100 lbs. of stock of fat about 10 to 12 lbs. of caustic-soda lye of 38° B., also reduced by the addition of water to 25° B. The boiling process is the same as for every other kind of soft soap, but careful light fitting is necessary so that the soap will not turn out slippery and short; it should also not be too rich in carbonate, as otherwise it becomes wet.

*White silver soap.* Special efforts are at present made to produce a particularly white silver soap, and it is scarcely necessary to mention that this is possible only by the use of suitable very pale materials, prime refined white American cotton-seed oil, best quality of tallow and, for better combination, some pale palm-kernel oil being above all required for a snow-white product. Besides, special attention has to be paid to the content of moisture, proportion of causticity and fitting

in order to be successful in turning out the soap smooth and delicate in accordance with the stock of fat.

An excellent stock for such soap consists of prime white American cotton-seed oil 390 lbs., white tallow 190 lbs., pale palm-kernel oil 20 lbs.. The process of manufacture is as follows: The lye required for boiling is best prepared from potash lye of 50° B., to every 100 lbs. of which are added for the necessary reduction of causticity about 25 lbs. of refined 96 to 98 per cent. potash dissolved in water. The lye is generally brought to 25° B., and for the saponification of the above-mentioned stock about 780 lbs. of such lye, as well as about 120 lbs. of caustic soda lye of 25° B., are required.

Boiling is effected in the usual manner by bringing the stock of fat and oil into the kettle together with about 200 lbs., of potash lye of 25° B. and about 70 lbs., of water and quickly bringing about combination over a moderate fire. When combination has been effected, add gradually but quickly, to prevent the mass from thickening, about 500 lbs. more of potash lye of 25° B., maintaining the mass at a moderate boil. The soda lye is then also added and incorporated by boiling, when by a further addition of potash lye of 25° B. the soap will soon come to a clear paste. By more vigorous boiling the excess of water will then be soon evaporated and the soap be clearer, thicker and shorter. The froth upon the soap will gradually disappear and the latter will commence to boil in larger plates and audibly break into roses.

When the soap has been evaporated so that it is free from froth, fitting is proceeded with. Samples of the finished soap when dropped upon the glass should pile up slightly, show a faintly perceptible flower, remain for some time liquid in the center, be sufficiently firm and short, become white only after some time, and adhere firmly to the glass. If the samples show the above mentioned properties, the soap may be considered of good quality; otherwise corrections such as may be necessary have to be made. The finished soap is brought into clear dry vessels in which when thoroughly cooled and after

storing for some time, it fully matures and acquires the white silvery appearance.

*Smooth olein soap*.—In winter this soap may be prepared from cottonseed-oil alone; in summer it is, however, necessary to use in connection with the oil some solid or semi-solid fat together with more soda lye, as otherwise the soap will when kept become more clear and lose its silvery appearance.

The process of boiling is generally as follows: 1000 pounds of refined cotton-seed oil and about 400 pounds of potash lye of  $16^{\circ}$  to  $17^{\circ}$  B. are brought into the kettle and combined over a moderate fire. Combination being established add 500 pounds of lye of  $18^{\circ}$  B. to the mass and increase the fire. Then with constant boiling and vigorous firing, add 600 pounds of potash lye of  $24^{\circ}$  B. in portions of 200 to 300 pounds each and 250 pounds of soda lye, whereby a clear paste will be formed, which is freed from superfluous water by thorough boiling. The mass now becomes thicker, clearer, and shorter, the froth upon the surface gradually disappears, and the soap boils in plates, runs from the paddle in raised streaks without spinning threads or breaking off, and audibly breaks into roses. During the boiling 40 to 50 pounds of potash solution of  $24^{\circ}$  B. are generally added which renders the soap clearer and smoother, and it is also fitted as much as possible with potash solution while it breaks into roses. When properly fitted a sample upon the glass must show flower and otherwise be clear, sufficiently short, and firm. The sample being thoroughly cooled, a white ring with thread-like, radiated crystallization extending towards the center must first show itself in the still transparent soap. At the same time a star-like crystallization forms in the center, which extending outward soon grows through the entire sample and gives it the lustrous, mother-of-pearl appearance. The same process of crystallization takes place later on in the barrels.

If the sample upon the glass becomes, however, white at once and slippery and can be moved, the soap is too strong and oil must be added until the sample shows a slighter flower

and becomes white and lustrous only after cooling. If, on the other hand, the sample shows only a slight sign of flower and does not keep clear until cold, but is whitish turbid in the center, the soap is too weak, which is remedied by carefully adding lye of 25° B. and eventually potash solution.

When the fitted soap breaks into roses and appears sufficiently short, evaporation is discontinued and the fire removed. The soap remains standing over night in the covered kettle. The next morning the froth formed upon the surface is removed and large samples are laid aside to cool. The behavior of these samples indicates whether the soap is of good quality in all respects, or whether it can be improved by crutching in strong potash lye or potash solution. Such corrections must, however, be done very carefully, as with an addition of too strong potash solution the soap becomes too white, and with potash lye too firm. The soap is finally perfumed with oil of mirbane to cover its not very agreeable odor, and then brought into barrels.

Still more advantageous than the use of cottonseed oil for the manufacture of smooth olein or silver soap is that of peanut oil. With the use of smaller quantities of soda lye, soap from cottonseed oil requires a longer time for complete crystallization before the silvery soap can be brought into the market. With the addition of a large quantity of soda the soap, to be sure, becomes white more rapidly, but also too firm and short, so that it separates lye. With peanut oil the case is different, as the soap prepared from it acquires the white, mother-of-pearl appearance with a small addition of soda lye, and is also smoother and whiter than that made from cottonseed oil.

A white soap of prime quality from tallow and 10 per cent. palm-kernel oil, the preparation of which differs somewhat from the previous one, may here be mentioned. Potash lye only can be used. The soap must also be kept quite carbonated, and with the use of potash lye of 50° B. for every 100 pounds of it about 32 to 33 pounds of refined 96 to 98 per cent. potash are required for the reduction of causticity. The

soap is only evaporated to the point of breaking into roses and then fitted to a good flower. Boiling is effected in the same way as for other soft soaps. Moderate flour-filling—5 parts flour, 10 parts potash solution of 12° B. and about 5 parts potash lye of 28° B. for fitting—is of advantage with this soap. The soap thus prepared is brought into barrels and worked through the next day; it is remarkably white, but does not show a mother-of-pearl appearance.

*Turpentine-sal ammoniac soft soap.*—The soap brought into commerce under this name is generally made from cotton-seed oil, peanut oil, horse fat, hog-fat, tallow, etc. in varying proportions, and when cooled to about 167° F., a quantity of turpentine and ammonia, or in place of the latter, solution of ammonium sulphate is crutched in.

The lye for this soap is prepared in the same way as for other soft soaps. Proportion of lime in summer 40 to 42 lbs. for 100 lbs. of 96 to 98 per cent. potash, and in winter 38 to 40 lbs. During the warm season of the year about 20 to 25 per cent. of soda lye is also used and during the cold season from 10 to 16 per cent.

The soap is as a rule prepared as follows: Cottonseed oil 450 lbs. and pale horse fat or hog fat 450 lbs. are brought together with a portion (about  $\frac{1}{3}$ ) of the required lye of 18° to 19° B. into the kettle, and combined, whilst crutching, over a moderate fire. The soap is then boiled clear, more lye of 27° to 28° B. being added and the fire increased, and it is at the same time properly fitted with potash solution of 24° B. With the use of much horse fat which, as a rule, is quite rancid, the soap frequently becomes somewhat thick, sluggish and viscid, and to bring it to boiling regularly the addition of a larger quantity of potash solution of 24° B. will be required; by this process the yield will also be increased and a more delicate and whiter product obtained. When the soap boils easily in large plates, audibly breaks into roses and appears clear and short, fitting to flower and lye-ring is proceeded with. A sample of the finished soap dropped upon the

glass should be clear, sufficiently short and firm, consequently show perceptible flower and only after some time become white. Fitting is here also of great importance; soap fitted too weak becomes short, when stored and when fitted too strong, slippery and separates lye.

By using, in place of tank-lye, lye of 32° B. from 100 per cent. caustic potash the following stock could be employed: Cotton-seed oil 450 lbs., pale horse fat or hog fat 450 lbs., caustic potash lye of 32° B. about 600 lbs., caustic soda lye of 37° B. about 75 lbs., as well as about 70 lbs. refined 96 to 98 per cent. potash in solution, 3 lbs. potassium chloride and 18 lbs. crystal soda for the reduction of causticity. The commercial potash lye of 32° to 33° B. and the caustic soda lye are, as a rule, reduced by the addition of water, to 24° or 25° B., the potash required being treated in the same manner.

Boiling is generally effected as follows: Bring the stock of fat and oil into the kettle, add about one-third of the caustic lye, together with a portion of the potash solution, and effect thorough combination over a moderate fire. While the mass is boiling easily, quickly add alternately caustic lye and potash solution till the soap boils in a clear paste. Continue in this way till the soap, after sufficient evaporation, has the necessary fitting and is finished.

As a rule this soap is ground with solution of potassium chloride or potash, a yield of about 250 per cent. being thus obtained. For augmenting the soap, 5 to 10 per cent. of flour is also frequently added, there being, for instance, used for fitting, flour 6 parts, potash solution of 10° to 12° B. 12 parts, silicate of potash 4 parts and caustic-potash lye of 28° B. about 6½ parts.

When the soap has sufficiently cooled crutch in for every 100 lbs. of stock of fat or oil about 2½ lbs. of turpentine and ½ lb. of ammonia or, in place of the latter, ½ lb. of ammonium sulphate dissolved in 2 lbs. of water; bring the soap into barrels and cover the latter. The addition of ammonia or ammonia salts would appear to be useless, as the ammonia formed soon volatilizes.

For the preparation of this soap with caustic potash lye of 50° B., for every 100 lbs. of the latter about 25 lbs. of refined 96 to 98 per cent. potash are required for reduction of causticity, and for filled soaps about 18 to 20 lbs. The reduced lye obtained is brought to 25° B., and 150 lbs. of it are required for the saponification of 100 lbs. of stock of fat and oil. For every 100 lbs. of stock of fat about 10 to 12 lbs. of caustic soda lye of 38° B., reduced to 25° B. by the addition of water, are also used. The process of boiling is the same as previously described.

*White soft soap.*—Under the name of *bleaching soap* or *white soft soap* a product is brought into commerce which is a soft soap only in name and consistency. It is generally prepared by boiling 100 pounds of cocoanut oil to a paste quite free from froth with about 200 pounds of soda and potash lye of 20° to 21° B., fitting strongly, and crutting into the paste 500 to 600 pounds heated silicate of soda previously mixed with 50 to 60 pounds of lye of 20° B. The mass is perfumed with oil of mirbane or lavender oil, and finally stirred until cold. Palm-kernel oil and bleached palm oil are frequently substituted for a portion of the cocoanut oil.

In Southern Germany a so-called tallow soft soap is prepared by heating 100 pounds of tallow with 250 pounds of caustic soda lye of 7° B. over a moderate fire until good combination is established, then adding successively 250 pounds of caustic soda lye of 3° B. and heating the mass, without, however, allowing it to boil, until it is quite clear. The soap, after resting for some time, is perfumed with oil of mirbane and poured into barrels.

*Filling of soft soaps.*—For the purpose of artificially increasing the yield of soft soap it is first of all necessary that it should have been correctly boiled and fitted, *i. e.*, that complete saponification of the fats by the alkalies has taken place. Further, the soap to be filled must be sufficiently evaporated, so that it can better absorb and hold the filling material. And as most filling materials possess shortening properties,

the soap must also stand "well in lime", *i. e.*, somewhat more caustic lye must be used in boiling. Only a compact, sound basis-soap (with not too high a content of rosin) is capable of absorbing a suitable filling material.

As the soapboiler can never know in advance the quantity of filling material the soap may be able to absorb without suffering in consistency and appearance, it is advisable to determine the amount by a test with a small quantity of finished soap. The soap is generally filled, when cooled to about 167° to 178° F., the materials chiefly used at the present time being potassium chloride, "gelatine," water-glass, "sapolite," potash filling, filling lye, and, where the appearance of the soap permits, potato-flour. The smooth, transparent and artificial fig soaps being the ones chiefly filled, the filling materials used and their behavior are here given:

For filling these soaps, potassium chloride is perhaps used more than any other material. Shortly before use, it is dissolved in water, and the solution set at 13° to 14° B. Of this solution, 20 to 25 per cent. of the fat used is crutched into the finished soap cooled to about 167° F., and the latter is eventually again fitted with strong caustic lye. Soaps filled with solution of potassium chloride are capable of vigorously resisting the cold in winter, and, with a very much reduced proportion of soda, do not need too strong a fit.

"Gelatine," brought into commerce by several manufacturers, is also much used for filling these soaps, 20 to 25 per cent. being crutched into the finished product, without a further fitting being required. As the "gelatine" is prepared chiefly from silicate of soda, it is advisable to make a corresponding reduction in the proportion of rosin and soda.

The so-called "sapolite," brought into commerce by a firm in Offenbach, Germany, is nothing but a mixture of potassium chloride and sodium sulphate. It is used in the following manner: Dissolve 1 part of sapolite in 2 parts of hot potash lye of 6° B., and add 2½ to 3 parts of water. The resulting clear solution should show about 16° B. (in winter 20° to 22°).

About 20 to 25 per cent. of the oil used of the solution is then crutched into the cooled soap. Soap thus filled has a beautiful, clear appearance, but as a sample upon glass must show a vigorous flower, subsequent fitting is sometimes required. A reduction of the proportions of rosin and soda is also recommended with the use of this filling material.

There are numerous receipts for the preparation of the so-called filling lye; two very good ones are:—

1. Saponify 50 pounds of cocanut-oil with 100 pounds of soda lye of 20° B., and crutch into the resulting soap 200 pounds of potassium chloride and 100 pounds of soda-ash dissolved in 2000 pounds of water.

2. Dissolve 100 pounds of semi-grained or grained soap in 600 pounds of hot water, and crutch into the solution 100 pounds of solution of potassium chloride of 15° B. and 150 pounds of silicate of soda of 38° B.

About 20 per cent. of the cold filling-lye may be added to the cooled soap, which must however, be again fitted.

Potato-flour is to a considerable extent used for filling soft soaps. It does not swell much either in cold water or in an alkaline solution, but when caustic lye is added it is at once converted into a smooth, stiff paste. It possesses also the property of fixing much moisture and making soap filled with it more compact and durable. As, however, the resulting product is more turbid only inferior qualities of soap are filled with potato-flour.

Filling with this material is effected as follows: A large vat of soft wood is placed near the kettle for the reception of the filling material. Pour into the vat 1 part of water or potash solution of 5° to 8° B., stir in 1 part of potato-flour and finally crutch in 1 part of silicate of soda. The latter may be omitted using in place of it 1 part of potato-flour stirred into 2 parts of potash solution of 10° to 12° B., or into potassium chloride solution. Add to the filling material thus prepared, whilst stirring vigorously, a sufficient quantity of the finished soap from the kettle to form a dissolved creamy mass and then

thoroughly crutch it into the soap in the kettle. The soap, after the filling has been added becomes soft, viscid and long, and has again to be fitted or shortened, this being effected with caustic lye of 27° to 30° B. Add of this lye, whilst crutching vigorously, a sufficient quantity to impart the shortness required and a sample upon the glass appears full and round as well as firm and shows the required sharpness; for 1 part potato-flour about 1 part of caustic potash lye is generally allowed for fitting. The operation of filling is frequently, also carried on as follows: About the quantity of lye required for fitting is crutched into the finished soap previous to adding the filling material, the soap acquiring thereby an almost torn appearance. The filling, however is more readily and better absorbed, and the soap presents a more uniformly smooth appearance.

Natural fig soap is at present also somewhat filled, the principal materials used for this purpose being prepared silicate of potash, potash filling and potato-flour. With the use of the latter the soap must have a considerable content of water, as soap thus filled, if deficient in water, readily effloresces notwithstanding correct filling; when fitted too slightly the basis-soap becomes soft and syrupy.

Smooth olein or silver soap for domestic use is also frequently filled, potato-flour being generally used for the purpose. Soap thus filled should only be lightly fitted.

## CHAPTER XV.

### TEXTILE SOAPS.

In the textile trade various kinds of soap are used. They may be divided into two principal classes—hard and soft soaps—the former being almost exclusively soda soaps and the latter chiefly pure potash soaps without much filling, at least without any kind of after-filling or similar filling materials. The hard soaps used are chiefly the better kinds of grained soaps and are prepared partly upon spent lye and partly as settled soaps. Semi-grained soaps are occasionally used, while paste-soaps are almost entirely excluded; silicate of soda soap is sometimes employed, but only in the linen industry.

The work that soap has to do in the textile trade and the objects for which it is employed vary very much, a perfectly neutral soap being, for instance, required in dyeing silk, while in other branches an alkaline soap is demanded and in some, one with a fixed content of fatty acid. In one case a hard soap is demanded that when dissolved—generally 1 part of it in 8 to 9 parts of water—yields a jelly-like mass, in another, a soap that when dissolved forms a semi-solid dressing, and again one that dissolves to water-thin dressing. It will thus be seen that for the manufacture of textile soaps not only a thorough knowledge of boiling soap is required, but it must also be accurately understood for what purpose the soap is to serve, how it is to be used, and what properties it must possess. In order to furnish the proper kinds of soap the soap-boiler must know how to make up the stock of fats. If, for instance, a textile plant uses a dark milling soap from oleic acid, bone fat and fuller's fat, this soap, notwithstanding its unsightliness, may be an excellent neutral soap and when dissolved in 8 to 9

parts of water, yield such a thin dressing as is required. If a manufacturer knowing nothing regarding the preparation of textile soaps would furnish, in order to get trade, as a substitute, a pale tallow grained soap or a similar product of much better appearance, it would prove a decided failure as his soap being unfit for the purpose would not be used the second time, because, when dissolved, it would yield a thick, jelly-like dressing instead of a thin one.

For a soap that is to yield a jelly-like dressing, animal fats containing stearin should only be worked; the more stearin they contain the stiffer the solution will be. If in addition to palmkernel oil, cocoanut oil, cottonseed oil, etc., a portion of fats containing stearin is saponified, the resulting soap when dissolved will yield a semi-thick dressing. On the other hand, soap made only from palmkernel or cocoanut oil, with additions of oleic acid, cottonseed oil, peanut oil, olive oil, etc., will dissolve to a thin dressing. Soft soaps, such as potash soaps only yield when dissolved, water-thin dressings, which remain quite thin when cold.

Although it is frequently asserted by manufacturers and chemists that potassium oleate—soft soap—exerts a more advantageous effect upon fabrics and tissues than sodium oleate—hard soda soap—and that soft soaps are also more convenient for many purposes, neutral grained soaps, especially such as yield, when dissolved, a thin dressing, are mostly used for fabrics with sensitive colors. To meet the demands of the consumer such soaps must have a large content of fatty acid and be as neutral and free from odor as possible.

To comply with these requirements quite substantial fats have to be used for textile soaps, tallow, cocoanut oil, palmkernel oil, oleic acid and olive oil being especially employed. Absolutely neutral soaps are most readily made from olive oil and oleic acid, with additions of cottonseed and peanut oils besides palmkernel oil. Olive oil or oleic acid will, as a rule, be used as the principal fat for soaps that are to yield a liquid dressing. It is, however, not always an absolute condition

since for neutral olive-oil soaps foots containing stearin, and the green sulphur olive oils are also worked, while perfectly pure olive-oil soaps are only made in rare cases, because the consumer is as a rule not willing to pay the price they should bring. The manufacture of these actually neutral textile soaps requires thorough knowledge and great attention, as well as very precise work. Smooth white grained soaps quite neutral may, however, be made from palm-kernel oil, tallow, cotton-seed oil, etc.

It has lately become more and more the custom for soap manufacturers to guarantee the content of fatty acid in the textile soaps made by them, and as many firms requiring large amounts only buy on this basis, this has to be taken into consideration. In order to obtain a product satisfactory in every respect the soap-boiler must know how such a soap has to be boiled and what kinds of fat and alkalies agree with the fabric and color. The content of fat can only be determined from the finished product, there being no rule for establishing it while the soap is being boiled. If the content of fatty acid is too low, the soap has to be dried out till the desired content is attained. For neutral grained soaps consumers take for granted a content of fat of at least 62 per cent., and some of them demand a much larger one; in the latter case the object is attained by drying out the soap.

For the production of neutral soaps the exact saponification of the fats is above all necessary, as this can never be completely attained by subsequent preparation or liquefaction of the soaps. Neutral means that the fats must be so exactly saponified that neither free alkali or unsaponified fat is present in the soap. From this it will be seen that the neutral textile soaps are products of the finest quality. They are usually prepared upon paste precipitate like settled grained soaps, but with exact and careful work may also be prepared by boiling them to a finish upon pasty spent lye.

## 1. HARD SOAPS.

*Neutral olive oil soap.*—*Stock*: Olive oil 1000 lbs., cottonseed oil 500 lbs., palm-kernel oil 500 lbs.

This is a serviceable stock, the soap obtained from it answering all demands of textile manufacturers and replacing pure olive oil soap. Neutral soaps are most readily obtained by boiling in an entirely indirect way as follows:

For the saponification of the above-mentioned stock about 2000 lbs. of pure caustic soda lye of 25° B. are required. A portion of the lye, about  $\frac{1}{3}$ , is at once brought into the kettle together with the fat and combines quite well with the latter while melting. The soap is allowed to boil up high and when intimate combination is established more lye is added. No more lye is added until froth commences to form on the surface and puffing up from below is noticed, which is always an indication of thorough combination. The remainder of the lye is then gradually added in three or four portions. With palmkernel oil as a constituent of the stock and boiling with pure caustic lye, such energetic combination generally takes place after half the lye has been boiled with the fat that the soap is inclined towards becoming thick. To effectively prevent this add gradually to the soap while boiling enough dry common salt to restore normal easy boiling. During this adjustment the addition of lye can of course be continued with.

Resultant fitting is indicated by a perfectly clear paste, biting sensation upon the tongue, and finger-pressure. The soap is then allowed to boil for one hour in paste for intimate saponification when it is salted out till a fine round grain free from froth is in the kettle and the spent lye runs perfectly clear from the paddle. Any scrap and paste-grain on hand may now be added and the whole melted over a moderate fire, best over night.

The next morning the spent lye is removed and the soap ground with boiling water, till a thin paste has been formed, which, when thrown with the paddle, flies and possesses a readily melting finger-pressure. While grinding, fitting may

again be controlled ; the soap should produce a faint biting sensation upon the tongue. The kettle is finally covered for 48 hours for the soap to settle, when the clear soap is brought into small iron frames holding from 12 to 15 cwts. and allowed to cool. If boiling is done carefully and correctly a perfectly neutral soap is obtained by this quite simple method.

*Neutral white wax soap.* *Stock.*—Palm-kernel oil or cocoanut oil 1000 pounds, pale cottonseed oil 500 pounds, tallow or tallowy fats 500 pounds.

With this stock, one-half of which consists of palm-kernel or cocoanut oil, the direct method of boiling may be employed, a neutral soap being obtained if the operation is correctly carried on. About 1700 pounds of caustic soda lye of 30° B. are required for saponification. Bring the lye into the kettle, add any scrap on hand, and melt over a moderate fire. Then introduce into the kettle the stock of fat with the exception of about 100 pounds of palm-kernel oil, which are retained for fitting, and effect combination with the assistance of the crutch over a moderate fire or by steam. Curb to prevent boiling over, and without hesitation sprinkle 50 to 100 pounds of brine of 8° to 10° B. over the mass. When a thoroughly combined soap is in the kettle, test it with the tongue and fit it with the palm-kernel oil kept in reserve so that it produces quite a biting sensation upon the tongue ; should fitting lye be wanting, add the necessary quantity.

If, after having been correctly fitted, the soap boils through uniformly it will, after some evaporation, soon present the appearance of a quite finished semi-grained soap, and the precipitation of the paste with the assistance of brine can immediately be proceeded with. Too vigorous evaporation is not necessary ; it being sufficient if the soap boils quite thick and free from froth. If too much evaporated it frequently shows separation without the addition of brine, and then becomes thin and readily frothy. Good appearance and proper neutrality depend chiefly on correct precipitation and this has to be done with the greatest care. The work should very carefully be

done with weaker brine of  $18^{\circ}$  to  $20^{\circ}$  B., Dry common salt should never be used, as it dissolves too slowly, lies more on the bottom and is not so rapidly and uniformly distributed in the soap, so that too much of it may easily be added when it is not required. While salting out it is advisable to keep up only a very moderate fire, it being sufficient for the paste which is forming to break through only here and there. It has to be accurately determined from the quality of the paste and from the soap itself whether enough, too much, or too little brine has been used. A small portion of brine does not in the first instance, bring about a perceptible change in a well combined soap which already contains much salt derived from scrap and paste-grain; if more is added a thin, more grainy wax-like soap contracts in the center till it covers the entire kettle and only allows the dark paste, formed with a moderate fire, to break through here and there on the edge. Sufficient brine has been used when the soap has become thin and fit for the separation of the paste, showing at the same time a still somewhat moist finger-pressure. The fire can then be removed. When the soap has quieted down from boiling, the kettle is covered and the soap allowed to settle for 36 to 48 hours, or it is brought, after resting for two hours, into large frames.

*Green olive-oil soap from sulphur oil.*—For the manufacture of olive-oil soaps the better qualities of pale olive oil are less frequently used than olive oil foots, and especially the green sulphur oils, which are obtained by extracting the press-cake with carbon disulphide. The color of the oil thus obtained varies very much, there being found in commerce pale grass-green varieties, and also some very dark ones, mixed with much water and dirt. However, generally speaking, these oils purify themselves very readily during saponification after salting out. While all vegetable and other impurities easily sink to the bottom, something of the extracting agent remains in the soap, which imparts to the finished product a disagreeable odor if it is not preferred to boil the soap to a finish upon several

waters. Sulphur oil is frequently rather expensive; in this case suitable fats, such as cottonseed, peanut and sunflower oils may be used in connection with the oil, as well as some animal lardaceous fat.

The soap is not prepared like settled neutral grained soap upon paste precipitate, but is finished upon pasty spent lye, a firmer product being thereby obtained and very little grain from the paste-precipitate.

No matter whether the stock consists of sulphur oil alone or of a mixture of fats, the process of manufacture differs always from that of ordinary neutral soaps, because sulphur oil contains much free fatty acid and is therefore inclined to extraordinarily rapid combination.

Suppose the stock consists of: Sulphur oil 1000 lbs. cotton-seed oil 500 lbs., horse fat 200 lbs.

For the saponification of this stock about 1700 lbs. of not entirely caustic soda lye of 25° B. are required. Bring the lye into the kettle, start the fire or bring it to a boil and then add 50 lbs. of common salt, the object of this addition being to prevent too vigorous combination. When the salt is melted gradually add in portions the sulphur oil to the boiling lye. Saponification takes place while the mass rises and effervesces, and sometimes lumps are formed. Should the latter happen the lumps have to be disintegrated by boiling till a smooth grain is formed, when another portion of sulphur oil may be added. Continue in this manner till all the sulphur oil has been introduced and a fine-grained soap boils in the kettle. Now add the cottonseed oil and finally the horse fat. When nearly all the cottonseed oil has been introduced a slight combination will result and may be maintained. Even if there is no combination, it does not matter, as this first boiling is more of a purification. Test the soap with the tongue; if quite a perceptible biting sensation is produced, salt out till the spent lye is no longer jelly-like but runs from the paddle clear, though dirty. By allowing the covered kettle to stand for a few hours the colored lye settles and the spent lye is removed whilst stirring vigorously.

Boiling upon the second water is now proceeded with, the object of this being to again thoroughly purify the soap and to saponify any unsaponified fat still present. When the spent lye has been drawn off, add, whilst at the same time firing or introducing steam, 400 to 500 lbs. of caustic soda lye of  $4^{\circ}$  or  $5^{\circ}$  B. When a nice, thin paste boils up in the kettle fit again with lye of  $25^{\circ}$  B. so as to produce a biting sensation upon the tongue and salt out till the spent lye again runs off clear. Now add any scrap or paste-grain on hand and melt all over a moderate fire or with steam.

When everything is melted, the soap is ground by the addition of hot water till the spent lye runs slightly pasted from the paddle. The soap is allowed to settle for 36 hours, the kettle being covered, and is then brought into low frames. By this process firm soaps of fine quality which are almost entirely neutral can be made.

*Neutral olein textile soap.*—For this soap either saponified olein by itself, or combined with bone-fat or pale horse fat is used. The distilled olein now found in commerce is, if of good quality, also suitable for these soaps. Since it, however, contains less stearin than the saponified product it is advisable always to add a smaller or larger quantity of the above-mentioned animal fats.

The manufacture of olein grained soap is best effected by first bringing the quantity of lye required for the saponification of the fat into the kettle. Suppose a stock of 1200 lbs. olein and 300 lbs. horse fat is to be worked. Bring into the kettle 1500 lbs. of caustic soda lye of  $25^{\circ}$  B. together with about 50 lbs. of common salt, heat to boiling, and gradually add the olein. By this method uniform combination of the oleic acid is later on brought about and by the use of lye of the above-mentioned strength the formation of froth is prevented. If steam is available lye of  $28^{\circ}$  to  $30^{\circ}$  B. may even be used. When fitting has been carefully attended to, salting out is proceeded with till the spent lye runs off clear and a clear grain-free from froth is in the kettle. Any scrap on

hand is now added and melted, whilst the mass is gentle boiling; the spent lye is then allowed to settle and finally drawn off. For the production of a perfectly neutral soap it is advisable to boil upon a second water. For this purpose bring into the kettle after the spent lye has been drawn off, 300 to 400 lbs. of caustic soda lye of 3° to 4° B. and boil a thin clear paste soap which is again salted out after it has been fitted so as to produce a biting sensation upon the tongue. The grain is then gently boiled for one hour longer and the spent lye allowed to settle over night. The next morning the spent lye is drawn off, a few hundred pounds of weak caustic lye are again brought into the kettle and the mass is allowed to boil up, enough water being gradually added till a very thin paste boils in the kettle. The paste is then salted out till the spent lye runs off slightly pasty. When salting out has reached this point, the soap is again somewhat contracted by the addition of water. When finished the soap is allowed to settle for 36 hours in the covered kettle and then brought into small frames.

*Grained soap from fuller's fat.*—Fuller's fat can as pure fatty acid be saponified in a manner similar to olein or sulphur oil. It is generally used in connection with other fats, such as bone fat, horse fat, etc., for dark soaps, the resulting products being cheap textile soaps of good quality, suitable for many purposes.

The white olein found in commerce is usually distilled fuller's fat, and it is frequently used in connection with other fats for making textile soaps. However, for dark milling soaps the crude fuller's fat is as a rule used. Since it is generally very dirty and also contains water, it should first be boiled upon several waters; only the better, cleaner qualities may be boiled at the same time with the other fats, but in this case it is advisable to use at least two waters in the same way as described for olein soap.

A substantial soap of as good a quality as can be expected from such products is obtained from the following stock:

Fuller's fat 500 pounds, bone fat 500 pounds, caustic soda lye of 25° B. 1000 pounds.

When fuller's fat of actually good quality is available the stock may be composed of two thirds fuller's fat and one-third bone fat, but of very inferior grades only one-third can be used. For a soap which, when dissolved, is to yield a very thin dressing, somewhat more fuller's fat, or, still better, some olein, has to be used, and the following stock can be recommended : Fuller's fat 600 pounds, olein 400 pounds, bone fat 500 pounds. This stock is worked in the same way as the olein soap previously described.

*Grained soaps for heavy dressing.*—The soaps previously described yield, when dissolved in hot water, a water-thin dressing, while a heavy, jelly-like one is obtained by dissolving a grained tallow soap. These products are, as a rule, brought into commerce as mottled grained soaps. Three varieties of these soaps are chiefly made, namely, a perfectly pure grained tallow soap, one from a mixed stock of tallow and tallowy fats, and one from tallowy fats only. A grained soap from palm-kernel oil is also used in the textile industry, and as all these soaps are made in the same way, they will here be jointly discussed.

*Stocks. I. For pure grained tallow soap.*—Tallow 1500 pounds, caustic soda lye of 25° B. 1500 pounds.

*II. For grained tallow soap from a mixed stock.*—Tallow 750 pounds, bone fat or horse fat 750 pounds, caustic soda lye of 25° B. 1500 pounds.

*III. For grained palmkernel oil soap.*—Bleached palmkernel oil 750 lbs., tallowy fats 750 lbs., caustic soda lye of 25° B. 1500 lbs.

For soaps which are to dissolve with greater ease the mixed stock or that of palmkernel oil is used, while a soap that is to yield a very heavy dressing, can only be made from tallow. The process of boiling these soaps is quite simple, thorough and intimate combination being the first requirement and this, when working tallow, demands great attention. The

soap should also be of the proper quality to form a good flux in the frame.

When working with direct fire lye of 25° B. is too strong and even when boiling with steam it is advisable to add some water.

Suppose stock II is in the kettle, then add 100 lbs. of lye and 200 lbs. of water, or introduce direct steam and combine the mass by boiling. When thorough combination has been effected continue with the addition of lye and eventually of some water, frequently regulating the fitting and combination till there is a clear, thoroughly fitted paste. Proper fitting is of the utmost importance because tallow and tallowy fats saponify with great difficulty as many soap boilers have found out to their sorrow. It is often assumed that the soap has sufficient fitting, but when boiling is continued it will be noticed that it is still much too weak, and frequently so weak that it can only be protected from running together and thickening by the speedy addition of lye. For the less experienced soap boiler it will be advisable to allow these soaps to boil for a longer time in paste so as to convince himself that there is sufficient fitting and only then proceed with salting out till the spent lye runs off clear and a strong, round grain boils in the kettle. Now add the scrap on hand and melt it by moderate boiling. With this first salting out and boiling through, the soap, as a rule, will not be sufficiently strong and salt will still be lacking. Dry common salt is then added till a quite heavy soap boiling deep down in the kettle is produced. By now allowing the kettle to stand for half an hour a liquid grained soap, the thickness of a finger, should have formed on top, the heavy grain lying beneath it. If considerably more liquid soap has been formed, the soap is not firm enough and common salt has still to be added. If, on the other hand, the soap is too strong and no liquid soap has been formed, some water has to be added and the whole again boiled through. The finished soap is for two hours allowed to settle in the kettle and then brought into frames which should not be too

wide or too high and capable of holding 40 to 60 cwts. Before commencing to draw the sheaves the soap in the frame is worked through with long stirring paddles so that it becomes very smooth and the spent lye still adhering to it can better settle on the bottom. The operation of drawing the sheaves is effected as follows: An iron rod reaching to the bottom of the frame is pushed down at one end of the latter and lines as straight as possible are drawn, about  $\frac{1}{4}$  inch apart, first lengthwise and then crosswise through the entire frame. As the drawing of perfectly straight lines by hand is even with considerable practice next to impossible, the use of a machine constructed for the purpose is preferable.

*Smooth white textile soap.*—As a substitute for the mottled soap above described a cheaper smooth white grained soap is boiled to a finish direct upon spent lye from an inferior quality of tallow, horse fat, pale bone fat, and eventually also some benzine fat or glue fat. The manufacture of these soaps is quite simple if attention is paid to thorough and intimate saponification. A stock which can be recommended is as follows: Tallow 300 lbs., horse fat or bone fat 400 lbs., glue fat 100 lbs., benzine fat 200 lbs., caustic soda lye of  $25^{\circ}$  B., 1000 lbs.

Boiling is effected as follows: Bring the stock into the kettle, add 200 lbs. of lye and some water, start the fire or boil the mass with direct steam till a thoroughly combined paste has been formed. Then continue with the addition of lye and boil to a clear well-fitted paste and salt out the latter till the spent lye runs off clear. Now add the scrap, melt it over a moderate fire and allow the soap to rest for two hours for the spent lye to settle. Then remove the latter, add 300 to 400 lbs. of caustic soda lye of  $3^{\circ}$  to  $4^{\circ}$  B. and bring the soap into combination by boiling. While boiling add enough water so that the soap boils quite thin. When with intimate combination thorough fitting has been attended to, salt out carefully till the spent lye runs off just slightly pasty. If salting out has been carried too far contract the soap again by

adding water. The finished soap is allowed to settle for 24 hours in the well covered kettle and then brought into small frames to cool.

## 2. SOFT SOAPS (POTASH SOAPS) FOR THE TEXTILE INDUSTRY.

Under smooth soft soaps are understood products which are brought into commerce without the formation of figging and also those which are not transparent and clear but of salve-like consistency—the so-called silver soaps.

While, generally speaking, soft soaps are of less importance for the textile industry than hard soaps, some varieties are used to a considerable extent, natural fig soap from olein and tallow being most in demand. Besides there is a firm potash soap, the so-called economy soap, which is much used, chiefly, where sheep's wool is treated.

*Smooth olein soft soap.*—Saponified olein is the best material for these soaps, though the best grade of distilled olein also yields products of good quality. Dirty distilled olein of inferior quality is entirely unsuitable for the manufacture of soft soap.

In boiling the basis-soaps from olein some other suitable fats such as cottonseed oil, peanut oil, etc., may be added; linseed oil, however, should not be used for these textile soaps, because it gums and consequently is more difficult to remove from the fabrics. The operation of boiling the soap depends on the season of the year, and in summer a stock for textile soap requires an addition of soda lye, this being also partly necessary for the reason that these soaps are frequently sold under a guarantee of a certain content of fatty acid, at least 42 per cent being, as a rule, demanded, which corresponds to a yield of about 237 per cent.; this yield is obtained with the use of 20 per cent. of soda lye.

As caustic potash lye of 50° B. is at present available, these soaps can at all times be correctly prepared by adding from the start the required alkaline carbonate to the lye. For the manufacture of smooth olein soft soaps in summer, 12 pound potash with a content of 97 to 98 per cent. alkaline carbonat

are required for every 100 pounds of caustic potash lye of 50° B., and in winter 17 to 18 pounds. The proportion of soda lye has to be so regulated that in the spring and fall 5 to 15 per cent. of soda lye of 25° B. are worked for every 100 pounds of fat, and in midsummer 25 per cent.

The process of boiling is somewhat different from that of ordinary soft soaps, when neutral fats only are saponified. Saponification of a mixed stock consisting of: Olein 1000 pounds, cottonseed oil 200 pounds, soda lye of 25° B. 360 pounds, potash lye of 25° B. about 1440 pounds, is effected as follows:

Bring all the lye required for saponification into the kettle, start the fire or bring to a boil with direct steam, and then add the olein in portions. The latter is saponified, the mass rising in the kettle and lumps being frequently formed. Continue boiling till these lumps are disintegrated, then add another portion of olein, allow it to saponify, and continue with the addition of olein till all of it is in the kettle; then add the cottonseed oil. When nearly the entire stock of fat is in the kettle the soap passes into combination. It is then boiled until quite free from froth and fitted so as to show a good flower upon the glass. By now evaporating to a finish free from froth, so that the soap breaks into small roses over the entire surface, it will show a good flower upon the glass and the samples will pile up. If the samples cool without being viscid, the basis-soap is ready.

In case the soap is viscid some potash solution of 30° B. may be added to reduce the causticity. Should the soap, in consequence of this addition, become too strong, olein or cotton seed oil has to be added till the proper degree of fitting has been attained. Soap thus fitted and free from froth will give a yield of 233 to 240 per cent. If the soap is not to be sold according to the content of fatty acid, 15 to 20 per cent. of potassium chloride solution of 13° B. may without hesitation be crutched in the next morning, but the soap has to be again fitted to "flower."

*Smooth green sulphur oil soap.*—This soap is also in demand in some branches of the textile industry. It may be prepared from sulphur oil alone, or with the additions of olive oil foots containing stearin, or that of other fats such as cottonseed oil, peanut oil, etc.

The green sulphur oil is frequently very dirty and contains water. In this case it must, previous to saponification, be purified and clarified upon salt water. By thoroughly boiling upon the salt water it also loses some of its disagreeable odor. For the summer months sulphur oils containing stearin, and olive oil foots are very suitable, but for the winter months it is advisable to use some cottonseed or similar oil as an addition. If sulphur oil is more expensive than cottonseed oil or peanut oil, the latter may be used as principal oils, a pale soap of excellent quality being, for instance, obtained from  $\frac{2}{3}$  cottonseed oil and  $\frac{1}{3}$  sulphur oil.

In boiling these soaps it is always well to boil the sulphur oil by itself; it enters quickly into combination and the soap readily clods. By, however, bringing at once as much lye into the kettle as the oil requires for saponification and gradually crutching the oil into the hot lye such unpleasant occurrence is avoided.

Potash soap can also be made from olive oil foots but it is advisable to boil the oil upon salt water partly on account of its odor and partly for the precipitation of the coloring matter and particles of dirt.

Sulphur oil by itself or in connection with other oils is best boiled in the same manner as previously described for olein soft soap. If, however, a predominant quantity of cottonseed or peanut oil is used and only a small quantity of sulphur oil, the ordinary method of boiling customary for neutral fats is employed. With a stock of cottonseed oil 100 lbs., sulphur oil 250 lbs., the same proportion of causticity is used as when working with sulphur oil alone. In the spring and fall 5 to 15 per cent. of soda lye is taken and in midsummer 25 to 30 per cent., while in winter it is best to entirely omit the soda lye.

When boiling with caustic potash lye of 50° B. 12 pounds of 97 to 98 per cent. potash are used for every 100 pounds of caustic potash lye to reduce the causticity, and in winter 15 to 17 pounds; this gives the proper lye for boiling.

For the saponification of the above-mentioned stock about 1875 pounds of potash lye of 25° B. are required; in case 20 per cent. of soda lye is to be used together with the potash lye, the quantity of the latter has to be accordingly reduced. When boiling in the old-established way, 30 pounds of caustic potash lye of 25° B. and 300 pounds of water are added to the stock of fat in the kettle. Start the fire or boil with steam till combination is effected. Then add more potash lye of 25° B. till about two-thirds of it has been used. The soap has now sufficient lye to prevent it from readily becoming thick. Now work in the soda lye and fit with the remaining potash lye to a light flower upon the glass. The actual evaporation to a finish is only proceeded with when it is shown by a sample upon the glass that sufficient lye in proportion to fat has been added. The soap is then evaporated till it is free from froth, which finishes the operation. If the soap is to be filled, 20 per cent. of potassium chloride solution is crutched in the next day, and it is again thoroughly fitted.

*Natural fig soap from olein and tallow.*—There is considerable demand for this soap, which is boiled with potash lye alone. The operation of boiling is the same in summer and in winter, the only difference being in the proportion of causticity and stocks of fat. More tallow is used in the summer months and the soap must be made somewhat more caustic than in winter. When working with caustic potash lye of 50° B., add for every 100 pounds of it 20 pounds 97 to 98 per cent. potash, and in winter 30 pounds.

Saponified olein is the best material for these soaps, though a good quality of the distilled product may also be used.

*Stock for summer.*—Olein 1000 pounds, tallow 900 pounds, crude palm oil 100 pounds, caustic potash lye of 35° B. 3000 pounds.

*Stock for winter.*—Olein 1200 lbs., tallow 700 lbs., crude palm oil 100 lbs., caustic potash lye of 25° B. 3000 lbs.

The soap is made as follows: No matter whether working with steam or direct fire, the lye may always be used of the strength mentioned in the formulæ, because with olein in the stock the stronger lye always effects thorough combination. The palm oil in the stock is temporarily reserved and introduced later on, a soap of a finer color being thus obtained. Otherwise the entire stock may be brought into the kettle together with one-fifth of the caustic potash lye of 25° B. and, when the mass is boiling, combination will, with the assistance of the crutch, be immediately effected. The remainder of the lye is then gradually added, allowing the mass to thoroughly boil through after each addition before introducing the next portion, and the soap is then fitted so as to produce quite a biting sensation upon the tongue. It will now boil quite free from froth and vigorous evaporation will not be necessary. The palm oil is then introduced whilst the soap is boiling gently, and when thoroughly saponified, fitting to a light flower upon the glass is proceeded with, the soap being at the same time evaporated so that it is free from froth and bubbles, which is recognized by its breaking into roses.

When the last portion of the palm oil has been incorporated with the soap and the latter has been sufficiently fitted, it will be seen from the way it boils in the kettle, otherwise from samples, whether it has been evaporated too much or too little, and also whether it is too caustic or too rich in carbonate.

The most experienced soapboiler is frequently deceived regarding the quality of a strongly evaporated natural fig soap as the normal proportion of fitting cannot be recognized. A sample upon the test-glass should slowly cool and when cold on the sides allow of the finger being passed through the center; it should also not pile up too high. When the soap is entirely cold the fitting should perceptibly lie upon it like a breath.

In boiling natural fig soaps it is of course first of all neces-

sary to see that the potash and potash lye are perfectly free from soda, because on this depends the good flogging of the soap. The slightest content of soda causes the soap to become inert, or at least the formation of a torn feathery flogging. Soaps properly boiled may the next day be filled with some solution of potash or potassium chloride of 13° B. When not too much evaporated a yield of about 245 per cent. will be obtained.

*Economy soap.*—Besides the soft soaps previously described, there are made in some localities so-called economy soaps the best qualities of substantial fats and potash lye only being used for them. They are thus firm potash soaps from varying stocks of fats, the most suitable components being tallow, bone fat and palm oil, though small quantities of fuller's fat and wool fat are occasionally used, but the resulting product is of a darker color. This economy soap is a peculiar product, since, after being fitted with caustic potash lye, it receives in addition about 15 to 20 per cent. of potash solution of 30° B., and then has the appearance of overdone soft soap. It is then evaporated till a sample after lying for two hours upon the glass becomes firm.

The soapboiler who wants to manufacture this soap should understand why it is made in this way. Economy soap is chiefly employed in milling heavy woolen fabrics of sheep's wool. The latter contains fat, the so-called wool fat, which is never entirely removed by the preparatory treatment, and this has subsequently to be effected by means of soap, one with an excess of alkali being used. Now, if the soap should possess an excess of caustic alkali the fabrics and colors would be attacked and for this reason carbonate of potash solution is added which in milling the fabrics removes the particles of wool fat still adhering to them.

*Suitable stocks are:*—Tallow 500 lbs., bone fat 500 lbs., palm oil 100 lbs., caustic potash lye of 25° B. 1650 lbs., potash solution of 30° B. 220 lbs.;

*Or:*—Bone fat 800 lbs., tallow 200 lbs. wool fat or fuller's

fat 200 lbs., caustic potash lye of 25° B. 1800 lbs., potash solution of 30° B. 240 lbs.

The soap is made in the same way as soft soaps. The stock is brought into the kettle together with 250 lbs. of potash lye of 25° B. and 200 lbs. of water and the mass boiled till thoroughly combined. Then, to prevent the mass from thickening add again lye of 25° B. and continue doing so, whilst increasing the fire, till the soap has become quite clear and shows a light flower upon the glass. Now evaporate till the soap is quite free from froth and then add the potash solution. When all is thoroughly boiled through, it will be found that a sample upon the glass will become turbid like a thoroughly overdone soft soap. However, this does not matter, and should be so. In about two hours the soap again becomes clear. It is now evaporated till after lying for two hours it has become as firm as desired, *i. e.*, that, when cold, it can be cut into bars. The fire is then withdrawn, the soap allowed to stand till the next morning when it is thoroughly crutched and brought into frames to cool. The lye for boiling this soap is prepared from caustic potash lye of 50° B, by adding to every 100 lbs. of it 25 lbs. of 97 to 98 per cent. potash.

## CHAPTER XVI.

### WASHING POWDERS AND ALLIED PRODUCTS.

*Washing Powders.*—The term washing powders would seem the more correct one for this kind of detergents because the name soap powders, which is sometimes applied to them, may lead to confusion, as these products do not consist of soap alone, most of them containing, besides soap, free soda, generally in the form of soda crystals. Free caustic soda should not be present in them, or at least only traces of it.

The preparation of washing powders is comparatively quite simple, although various methods may be adopted to obtain a product of good quality.

1. A soap solution is boiled to a finish and to this is added, whilst constantly stirring, soda ash until a perfectly homogeneous mixture results. After cooling, the pasty mass is brought into frames, and when it has become hard is crushed and ground to a more or less fine powder.

2. Finished soap is with the addition of water liquefied to soap paste when the soda is introduced, the further operations being the same as given under 1. However, this method is of advantage only where waste and scrap are available or can be cheaply procured.

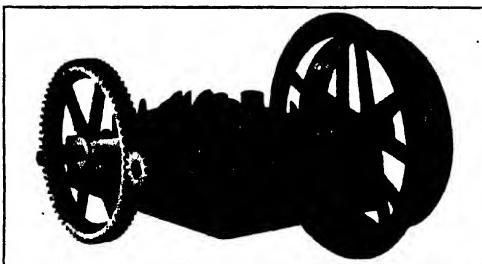
3. A fatty acid is melted together with soda so that a moist mass is formed, which is exposed in thin layers to the air; with a rise in the temperature a sort of saponification takes place, after which the mass is crushed, ground and sifted.

According to Method 1, a thin soap paste is boiled from suitable fats, sometimes with the addition of rosin. The soap while still liquid is brought into a jacketed pan furnished with a strong stirring apparatus so that the mass can be thoroughly

mixed. The soap paste in the pan should be thoroughly cooled by running cold water through the jacket. When the soap paste has been sufficiently cooled, the soda is slowly and regularly added, whilst constantly stirring, and without interrupting the cooling. When all is thoroughly dissolved and the mass is uniformly mixed, the soap is drawn off into frames to solidify.

The Blanchard soap powder crusher, manufactured by Houchin-Aiken Co., Brooklyn, N. Y. and shown in Fig. 35 with the hopper removed is a very useful machine. This crusher will take lumps of powder from the frame, in sizes not exceeding 12 inches thick by 20 inches wide by any length required, and reduce these large lumps to about the size of

FIG. 35.



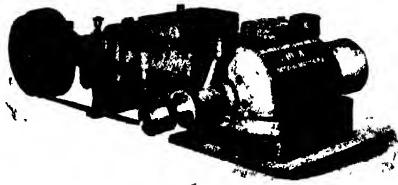
eggs or smaller, making an ideal product for feeding to the mill.

The usual practice is to tip the slab from the frame bottom to the floor and break it up with a pick or axe into a few large pieces, which can be easily thrown into the crusher. Where the powder is mixed on the floor or on trays, the crusher need not be used at all, but where floor space is valuable and the labor item expensive, the most economical method is believed to be to frame the powder and run it through the crusher. The stock can then be delivered into a conveyor and elevated directly from the crusher and stored in a bin over the mill, thus saving all handling of material.

Fig. 36 shows a Blanchard mill, also manufactured by the above mentioned firm. This machine is designed for the grinding of soap powder after leaving the crusher or from the drying trays. The powder is ground by repeated blows of rapidly revolving steel beaters.

A perforated screen made of a special wear-resisting metal is built into the machine, and all material fed into the mill is retained in the grinding chamber until fine enough to pass this screen. Therefore, the product, as it leaves the mill, is ground fine and ready for packing. The feeder is very simple and effective and is entirely automatic. It can be regulated to deliver any required quantity up to the capacity of the machine. This machine is intended to be supplied from a storage bin

FIG. 36.



overhead and discharge below into a receiving hopper or barrels. With this arrangement the labor cost is practically nothing, as it allows the attendant to devote his time to other work. To prevent the heating of the stock, the machine is provided with an effective means to maintain a sufficient circulation of air.

The operation, according to Method 2, is carried on in the same manner as Method 1, it being only necessary to convert the waste and scrap into a homogeneous paste into which the soda is mixed.

Method 3 is a very convenient one, since only fatty acids, especially oleines, are required and they can be saponified direct with sodium carbonate, it being then not necessary to

first prepare a soap paste, but simply to bring the soda solution into the mixing kettle, or melt the soda crystals and run in, whilst constantly stirring, the fatty acids. If required, a small quantity of caustic lye—10 to 12 per cent.—may be added at the end of the operation so as to be sure that all the fatty acid is saponified.

There is considerable variation in the composition of washing powders, they showing especially a great difference as regards the content of fatty acids, which are the most valuable of the separate constituents. Rosin may be substituted for a portion of the fatty acids, but for practical reasons too much of it cannot be used, as otherwise the resulting product would turn out smeary and not capable of being pulverized. Impure fats, such as bone fat, waste fat, etc., are sometimes used for the preparation of washing powders. In that case the soap has to be salted out before introducing the soda, the impurities then passing into the spent lye. Dark fats have to be bleached by adding a little chloride of lime solution. Free chlorine should not be present in washing powder, as the latter is not intended for a bleaching medium, and larger quantities of combinations with active chlorine would injure fabrics.

Many washing powders contain silicate of soda in the proportion of 2 to 5 per cent. of the total weight. While such an addition can do no harm, it is doubtful whether it is of any actual value, for the object of softening the water is to a sufficient degree attained by the soda alone. Mineral substances, such as talc, infusorial earth, etc., added to washing powders have a mechanically detergent action, but clothes washed with powders containing such additions have to be repeatedly and slowly rinsed to remove the mineral substance. Talc is especially added with the object of making the powder slippery so as to appear fat. Some washing powders are perfumed, generally with oil of mirbane or orris-root powder.

Good washing powders consist of: Fatty acid 30 to 40 per cent., soda 30 to 35 per cent., water 30 to 40 per cent. Inferior soap powders contain only 5 to 10 per cent. of fatty acid;

they are unfit for laundry work, but serve for scrubbing tables, floors, decks of steamers, etc. In the manufacture of inferior grades of washing powders all kinds of devices have been resorted to to make them appear more substantial. Thus, for instance, linseed meal has been boiled up direct with caustic lye, whereby a thin linseed-oil soap is obtained, and besides some albuminous substances pass from the seed into the solution. This produces quite a strong lather when the product is used and assists in the deception that it contains a larger content of soap than is actually the case.

*For washing electros and type* a soap powder has been introduced that contains very little fat, but from 10 to 15 per cent. caustic alkali. It is more convenient for use than caustic lye, but has the drawback of readily losing its content of caustic alkali, the latter being converted into carbonate by taking up carbonic acid from the air.

*Bleaching soda*, which has been widely advertised and is sold in considerable quantities, must also be classed as a washing powder. Notwithstanding its name, it possesses no bleaching properties. It varies very much in composition, but usually consists of soda crystals 80 parts, silicate of soda 20 parts.

*Thompson's washing powder* belongs to the better class of commercial washing powder. The following formula may serve as an example of its composition : Caustic soda 30 parts by weight, water 90 parts by weight (= 120 parts by weight of caustic soda lye of 30° B.), palmkernel oil 1000 parts by weight, oleic acid 50 parts by weight, soda ash 90 parts by weight, water 60 parts by weight.

Bring the caustic soda into a kettle, pour the water over it, and crutch occasionally until all is dissolved. Heat the lye of 30° B., which has thus been obtained, by a direct fire or steam to the boiling point, and then add, whilst crutching or stirring, the oleic acid which will immediately saponify. Next add the palmkernel oil and heat the mass until uniform saponification has taken place which is recognized by no uncombined fat or lye being noticed and by the appearance of a

homogeneous compact mass which on cooling becomes solid and produces a faint biting sensation upon the tongue. When the soap has been boiling through for about ten minutes add, whilst constantly agitating the mass, the quantity of soda which has in the meanwhile been prepared. The working in of the soda may, in the beginning of the operation, be done with a wooden stirrer, but finally a shovel or paddle has to be used. The more soda is used the more difficult the work becomes, because the soap mass, at first pasty, becomes doughy and finally crumbly. For working on a large scale a mixer or disintegrator is used for working the heavy mass of soap and soda ash. It is provided with a strong stirrer and sifting contrivance, and run by power. When working according to the first mentioned method, the hot, pulpy mass of soap is brought into low boxes or wooden frames in which in a few days it solidifies to a compact mass hard as stone. This mass has to be broken up, crushed and finally ground in the mill. With the use of the mixer or disintegrator the lumps of soap are broken up by being constantly worked so that the mass, assisted by self-heating, finally forms a powder which only requires sifting, the use of a mill being thus dispensed with. However, if a powder in the form of a fine flour is desired, it is advisable to pass the product through a mill. When working with direct steam, as is generally the case, about 60 parts by weight of water are brought into the mass by the steam as condensed steam so that a powder with a content of 35 to 36 per cent. fatty acid is obtained. Hence, the content of fatty acid may, as desired, be more or less reduced by the addition of soda or water. When working with indirect steam, or an open fire a powder with a content of about 39 to 40 per cent. fatty acid is obtained which may be reduced by the addition of soda ash and water.

*Washing powder with a high content of fatty acid* is prepared by heating in a kettle 200 lbs. caustic soda of 24° B., gradually introducing 150 lbs. oleic acid and 50 lbs. palmkernel oil, and boiling until a clear paste is formed. Then crutch in 200

lbs. soda ash and work the whole thoroughly through. Bring the thick pulpy mass into low frames to solidify. The mass when taken from the frames is broken up and ground to powder in the mill. The product thus obtained will keep perfectly dry in paper packages.

*Cheap washing-powders.*—Olein 140 lbs., palmkernel oil 52 lbs., unbleached palm oil 8 lbs. caustic soda lye of 25° B. 200 lbs.

Bring the lye into the kettle, bring it to the boil by steam or a fire, and successively add the fats. When the fat has saponified, combination and consequently a well-fitted paste is obtained. Now withdraw the fire or shut off the steam and, according to the quality of powder required, crutch in a larger or smaller quantity of soda ash until the mass becomes crumbly.

If powder of a very good quality is required, somewhat weaker lye is used. If, with the above mentioned quantities, about 120 lbs. of soda ash are added, a yield of 260 per cent., which corresponds to a content of 38 per cent. of fatty acid, is obtained. If the powder is to be filled with talc, stir up 2 parts of talc in 1 part of water and crutch in the mixture. If the powder, however, still contains much water, the talc may be stirred in dry. The more water is incorporated with the powder, the more soda can be added and the cheaper the product will consequently be.

Below a few formulas for washing powders are given :

I. Wax grained soap 200 lbs., or rosined grained soap 240 lbs., water 200 to 300 lbs., soda crystals 200 to 300 lbs., soda ash 160 to 200 lbs.

II. Rosined grained soap 200 lbs., soda crystals 800 to 1100 lbs., soda ash 200 to 300 lbs.

III. Olein 200 lbs., palm-kernel oil 140 lbs., crude palm oil 60 lbs., caustic soda lye of 25° B. 400 lbs., water 200 lbs., soda ash 2000 to 2400 lbs.

Crutch in the soda without allowing the mass to boil.

*Washing powder without the use of a mill.*—For small works

the preparation of washing powder without the assistance of a mill can be recommended, as the necessary utensils are on hand in every soap factory and it will at the utmost be necessary to procure a sieve. Suitable compositions are as follows.

- I. Soda crystals 200 lbs., soap 100 lbs., soda ash 100 lbs., or
- II. Olein 200 lbs., caustic soda lye of 25° B. 200 lbs., soda ash 200 lbs.

Bring into a small kettle the 200 pounds of crystal soda and melt in them the 100 pounds of soap previously finely crushed. When all is melted, stir in the soda ash and remove the fire. The process is exactly the same with composition II, which has first to be saponified with the lye. The fire is also withdrawn and the soda stirred in. In the meanwhile a number of trays are laid out in a cool room, and the contents of the kettle are spread out in thin layers upon the trays by means of a shovel. At short intervals the mass is turned with a shovel and at the same time broken up as much as possible by rubbing and cutting with the shovel, this operation being continued till the mass is so far cooled that it no longer smears but can be rubbed into small granules. A sieve is then placed over a large box and the mass rubbed through.

When working in accordance with the above-mentioned process the soap can be readily rubbed through the sieve without leaving behind any hard lumps which cannot be easily reduced. According to whether a fine or coarse sieve is used the resulting powder will be of a finer or coarser grain. It is advisable to rub the soap first through a coarse and then through a fine sieve, a uniform powder of fine quality being thereby obtained. A small percentage of soft soap may also be used in the preparation of this powder, it serving to render the product more delicate and more readily soluble.

*Dr. Link's washing powder* is also prepared without the assistance of a mill. It is an excellent product and its manufacture is very simple. If only olein and soda crystals are used in its preparation, an extremely delicate and fine washing agent is obtained which, when made without talc filling,

does not leave behind the slightest precipitate in washing.

The compositions of three different qualities are given below:

I. Ground or powdered crystal soda 160 lbs., olein 40 lbs., soda ash 28 lbs.

II. Ground crystal soda 176 lbs., olein 24 lbs., soda ash 20 lbs.

III. Ground crystal soda 180 lbs., olein 20 lbs., soda ash 16 lbs., talc 40 lbs.

Place two large cast-iron kettles alongside each other. Into one of them sift through a fine sieve the ground crystal soda together with the soda ash as well as the talc, if it is to be used, and mix all thoroughly. In the other kettle warm the olein and, if the powder is to be of a yellowish color, melt a small quantity of crude palm oil together with the olein.

Stir a portion of the mixed soda slowly into the fat using a large paddle till a doughy mass is formed. Then add gradually, but quickly, the rest of the soda and work the mass thoroughly through. When all is finely distributed rub the finished powder through a sieve into a large box, covering the latter, when full, with a lid. The mass becomes self-heated and passes through the saponification process. After a few days the powder can be filled into packages.

The whole operation is simple and easy. Care must especially be taken that no lumps are formed when the first portions of soda are added, as such lumps can be reduced only with great difficulty. A paste should first be formed which by the gradual addition of soda, whilst stirring thoroughly, finally gives a dry, doughy mass; by working in this way no lumps will be formed.

*Washing powder with silicate of soda.*—In preparing this powder first saponify in the semi-warm way the fat with the lye to a clear paste. Into this paste stir the silicate of soda solution, then immediately add the soda, and combine all by energetic crutching. Bring the pasty mass thus obtained, upon a smooth stone floor and work it with a shovel till it falls to pieces, and pass the latter first through a coarse, and

then through a fine sieve. The pieces obtained by the drying process may also be ground in a mill. Suitable stocks are as follows:

I. Palmkernel oil 150 lbs., olein 50 lbs., caustic soda lye of 25° B. 200 lbs., silicate of soda 150 lbs., mixed with soft water 380 lbs., and caustic soda lye of 38° B. 20 lbs., ammonia soda 50 lbs.

II. Olein 50 lbs., caustic soda lye of 25° B. 50 lbs., silicate of soda 250 lbs. mixed with water 100 lbs. and caustic soda lye of 25° B. 50 lbs., ammonia soda 500 lbs.

III. Palmkernel oil 80 lbs., olein 50 lbs., rosin 20 lbs., caustic soda lye of 25° B. 150 lbs., water 100 lbs., silicate of soda 200 lbs. mixed with caustic soda lye of 38° B. 30 lbs., ammonia soda 400 lbs.

*Disinfecting washing powder.*—The discoverer of this preparation, Mr. Hawliczek first prepares a mixture of ordinary commercial soft soap with carbolic acid or other phenols in suitable proportions. This mixture is heated until it acquires the consistency of a jelly. Infusorial earth is then mixed with this jelly till the mass becomes granular or pulverulent, whereby its cleansing properties are also increased. The following proportions have been found very suitable:

Soft soap 70 parts, carbolic acid 20 parts, infusorial earth 80 to 100 parts according to the desired quality of the final product. A small quantity of coloring matter, for instance, phenolphthalein may finally be added. The object of the addition of infusorial earth is, on the one hand, to transform the gelatinous mass into a solid form and, on the other, to retain the carbolic acid.

*Electra washing powder*—is composed of: Olein 3 parts, soda ash 53 parts, caustic soda 12 parts with water 32 parts.

*Gold dust washing powder* is said to be composed of: Anhydrous carbonate of soda 49 per cent., soap 43 per cent., water 8.1 per cent.

*Lobartes' washing powder contains*: Ammonia soda 48 parts, 75 to 80 per cent. potash 6 parts, 70 per cent. caustic soda 85

parts, silicate of soda 36 parts, potassium permanganate 1 part, thyme oil 0.5 part.

*Tine washing powder* is composed of : Carbonate of soda 58.47 parts, soap 26.10 parts, water 15.99 parts.

*Pearline washing powder* is said to consist of : Water 12.6 per cent., anhydrous carbonate of soda 32.3 per cent., soap 35.1 per cent.

*Scouring soap powder* is prepared by saponifying 30 parts olein, cocoanut oil or palmkernel oil with 36 parts caustic soda lye of 20° B. and crutting in 225 parts soda ash and 150 parts water. The mass, when cold, is ground to a fine powder.

*Soapine washing powder* is said to be composed of: Water 15.213 per cent., carbonate of soda 49.581 per cent., soap 35.205 per cent.

*Japanese washing powder* contains : Soap 12.0 parts, soda ash 66.0 parts, carbonate of soda 15.0 parts, ultramarine 0.3 part.

*Washing powders without soap*.—These products contain no fatty acid whatever, or at the utmost 1 to 4 per cent. An actual washing powder of good quality possesses the same advantages as soap. Even if it contains a large amount of soda it is equal to soap, provided its price corresponds to its content of fatty acid, and its action may perhaps be more economical than that of soap of equal value, because the salts of lime, alumina, magnesia and iron present in water are precipitated by the soda in the form of carbonates and not, at the expense of soap, as salts of fatty acids. This advantage cannot be claimed for many of the so-called washing powders, some of them having not even the value of pulverized soda as they are partly mixed with salts totally unfit for washing purposes. Some contain also heavy spar, talc and other mineral constituents. A few, however, possess considerable detergent power.

*Forster's washing powder*.—Borax 60 parts, crystal soda 20 parts, potash 10 parts, tartrate 6 parts, oxalic acid 5 parts, soluble blue  $\frac{1}{2}$  part.

The materials are intimately mixed and then form a bluish powder which is said to be very suitable for laundry use and

not to attack the ordinary colors of fabrics. When using this washing powder the clothes are first soaked in cold water, then soaped and allowed to lie in cold water. Water is then brought to the boil and after dissolving in it a quantity of the washing powder— $\frac{1}{2}$  ounce to 1 quart of water—the clothes are put in and allowed to boil for five minutes. Rubbing upon the wash-board or other manipulation is not required. The clothes are perfectly clean, all stains have disappeared, and all that is necessary is to rinse them.

*Palme's washing powder* is composed of: Borax 30 per cent., half-weathered soap 61 per cent., sodium chloride 4 per cent., corn or wheat starch 5 per cent.

*Washing powder for electros and type-setting machines.*—A white water-soluble powder sold for this purpose consists of: Saltpetre 7.20 per cent., soda 61.50 per cent., borax 12.62 per cent., water 18.68 per cent.

## CHAPTER XVII.

### TOILET SOAPS AND SOAP SPECIALTIES.

TOILET soaps are prepared essentially 1, by the cold saponification process; 2, by remelting and subsequent perfuming, and 3, by milling, *i. e.*, by comminuting, perfuming, coloring, and subsequently pressing together by means of specially constructed machines, so-called stock soaps carefully prepared for the purpose. Milled soaps are the most valuable and durable, and at the same time the most expensive, toilet soaps manufactured. They require a great deal of manipulation by costly machinery, which will be referred to later on.

*Toilet soaps by the cold saponification process.*—The manufacture of soaps by the cold process has already been described in Chapter XIII. A good quality of white, odorless soap prepared by cold saponification serves as basis-soap. For example,  $66\frac{2}{3}$  pounds of cocoanut oil are melted in a jacketed pan and after settling passed through a fine linen cloth or hair-sieve to remove any impurities. The oil is then run into the stirring kettle and when cooled to  $95^{\circ}$  F.,  $33\frac{1}{3}$  pounds of caustic soda lye of  $38^{\circ}$  B. are run into it in a thin stream with constant stirring until the oil is completely saponified and the mass commences to thicken. It is then cooled by stirring in the dissolved coloring matter, next perfumed, and finally brought into the frames, in which it is allowed to slowly congeal.

It may here again be mentioned that the fats used in the cold process must be as pure and fresh as possible and a good quality of caustic lye is absolutely necessary. The fats and lyes must be accurately weighed out, as an excess of lye in the soap makes it very hard, harsh, and biting. If, on the other

hand, too little lye is used, the fat would not be completely saponified, and the soap would be greasy and soon acquire a rancid odor.

Below some formulas for toilet soaps by the cold process are given:

*Eagle soap*.—Basis-soap 220 lbs., Neutral Brown 12,  $2\frac{1}{4}$  ozs., lavender oil 7 ozs., mirbane oil  $1\frac{1}{4}$  ozs., caraway oil  $6\frac{1}{2}$  ozs., orris-root oil  $8\frac{1}{2}$  ozs., musk tincture  $3\frac{1}{2}$  ozs., thyme oil and rosemary oil each 5 ozs.

*Bitter almond oil soap (genuine)*.—Saponify cocoanut oil 44 lbs and lard 66 lbs. with caustic soda lye of  $40^{\circ}$  B. 55 lbs. Perfume with bitter almond oil 7 to 12 ozs., bergamot oil  $3\frac{1}{2}$  to  $5\frac{1}{2}$  ozs., lemon oil  $1\frac{1}{2}$  to  $3\frac{1}{2}$  ozs.

Almond soaps are not colored. A peculiar kind of bitter almond oil soap is prepared as follows: Melt soap of a suitable quality and stir in almond meal. The resulting soap is perfectly opaque and when used for washing, or when only moistened, has a very agreeable odor of bitter almonds. This is due to the fact that bitter almonds contain a peculiar body—amygdalin—which on coming in contact with warm water is decomposed and yields several bodies, oil of bitter almonds being one of them.

*Marshmallow soaps*. A (*best quality*).—Mix 44 lbs. each of cocoanut oil, palm-kernel and tallow soaps. *Perfume*: Lemon oil  $3\frac{1}{2}$  ozs., bergamot oil  $1\frac{1}{4}$  ozs., lavender oil 14 ozs., neroli oil  $3\frac{1}{2}$  ozs., peppermint oil  $\frac{3}{4}$  ozs., verbena oil  $1\frac{1}{4}$  ozs., cinnamon oil  $\frac{1}{2}$  oz.

Marshmallow soaps are colored yellow or orange and bright red.

B (*medium quality*).—Soap stock and coloring matter same as for A. *Perfume*: Lavender oil 2 to 3 ozs., oil of cloves  $3\frac{1}{2}$  to 5 ozs., orange-peel oil 5 to  $8\frac{1}{2}$  ozs., patchouli oil 1 to  $1\frac{1}{2}$  ozs., cassia oil 2 to  $2\frac{1}{4}$  ozs.

C (*ordinary quality*).—Soap stock and coloring same as for A and B. *Perfume*: Lemon oil  $2\frac{1}{4}$  to  $4\frac{1}{4}$  ozs., caraway oil  $2\frac{1}{4}$  to  $3\frac{1}{2}$  ozs., curled mint oil  $1\frac{1}{2}$  to  $1\frac{1}{4}$  ozs., rosemary oil  $1\frac{1}{4}$  to 2 ozs., sage oil  $1\frac{1}{4}$  to 2 ozs., spike lavender oil  $7\frac{1}{2}$  to 10 ozs.

*Nutmeg soap.*—This soap with a pure nutmeg-oil odor is prepared by perfuming soap with optional quantities of nutmeg oil and coloring it, as a rule, yellow, saffron being used for the better qualities and curcuma for inferior ones. A still finer product is obtained by using for perfuming the soap, in addition to nutmeg oil, other suitable oils, for instance: White soap 220 lbs., nutmeg oil  $3\frac{1}{2}$  to  $5\frac{1}{2}$  ozs., mace oil  $\frac{1}{2}$  to 1 oz., oil of cloves  $\frac{1}{2}$  to  $\frac{3}{4}$  oz., tincture of musk  $\frac{1}{2}$  to  $\frac{3}{4}$  oz., cassia oil  $\frac{1}{2}$  to 1 oz.

*Honey soap.*—Cocoanut oil 100 lbs., caustic soda lye of  $38^{\circ}$  B. 50 lbs., water 2 lbs. Uranine orange  $\frac{1}{2}$  ozs., dissolved in boiling water; citronella oil,  $8\frac{1}{2}$  ozs., lemongrass oil  $1\frac{1}{2}$  ozs. peppermint oil  $\frac{1}{2}$  oz.

*Honey soap (filled).*—Cocoanut oil 100 lbs., caustic soda lye of  $38^{\circ}$  B. 50 lbs., talc 12 lbs., stirred into the warm oil before adding the lye. Uranine orange 2 ozs., dissolved in boiling water; citronella 7 ozs., cassia oil  $3\frac{1}{2}$  ozs., oil of cloves 1 oz.

*Bergamot soap.*—Cocoanut oil 50 lbs., tallow 50 lbs., caustic soda lye of  $37^{\circ}$  B. 50 lbs.—Brilliant soap green  $8\frac{1}{2}$  ozs. ground up in hot oil.

*Alpine flower soap.*—Cocoanut oil 50 lbs., tallow 25 lbs., lard 25 lbs., caustic soda lye of  $37^{\circ}$  B. 50 lbs.—Brilliant soap green  $8\frac{1}{2}$  ozs. ground up in hot oil.—Lemon oil  $2\frac{1}{2}$  ozs. lavender oil  $1\frac{1}{4}$  ozs., peppermint oil  $1\frac{1}{4}$  ozs., sage oil  $1\frac{1}{4}$  ozs., rosemary oil  $1\frac{1}{2}$  ozs., cinnamon oil  $\frac{1}{2}$  oz.

*Bismarck soap.*—Cocoanut oil 24 lbs., castor oil 4 lbs., soda lye of  $40^{\circ}$  B. 14 lbs.—Color dark with Bismark brown. Cinnamon oil  $2\frac{1}{2}$  ozs., oil of cloves 5 drachms, sassafras oil 8 drachms, bergamot oil 5 drachms, lemon oil  $1\frac{1}{2}$  ozs.

*Pumice soap.*—Ceylon cocoanut oil 100 lbs., caustic soda lye of  $38^{\circ}$  B. 52 lbs., glycerin 2 lbs., powdered pumice 40 lbs., water 4 lbs.—Frankfort black  $2\frac{1}{2}$  ozs., ultramarine blue 0.8 oz., bergamot oil 7 ozs., thyme oil 7 ozs. Add the color and pumice to the hot oil.

*Bouquet soap.*—Cocoanut oil 50 lbs., tallow and lard 25 lbs. each, caustic soda lye of  $37^{\circ}$  B. 50 lbs.—Windsor brown 0.8

oz.—Bergamot oil  $6\frac{1}{2}$  ozs., sassafras oil, oil of cloves and thyme oil  $1\frac{1}{2}$  ozs. each, oil of neroli 0.8 oz., tincture of musk 0.3 oz.

*China soap*.—Cocoanut oil 50 lbs., tallow 50 lbs., caustic soda lye of  $37^{\circ}$  B. 50 lbs.—Purple-red 0.8 oz.—Bergamot oil  $5\frac{1}{2}$  ozs., lavender oil  $3\frac{1}{2}$  ozs., palma-rosa oil  $2\frac{3}{4}$  ozs., lemon oil 2 ozs., cedar wood oil  $3\frac{1}{2}$  ozs., tincture of musk 0.3 oz.

*Glycerin cold-cream soap*.—Melt 25 lbs. of cocoanut-oil, 25 lbs. of tallow, 8 lbs. of castor-oil, and 2 lbs. of crude palm-oil, heat to  $167^{\circ}$  F., and dissolve in the mass 15 lbs. of scrap of white or yellow cocoanut-oil soap. Then stir in 30 lbs. of lye of  $36^{\circ}$  B. until the soap-mass becomes thick, and add 1 lb. of glycerin and 8 ozs. of spermaceti, the last previously heated to  $167^{\circ}$  F. Now cover the soap, and after allowing it to rest for half an hour crutch thoroughly. After again resting for  $1\frac{1}{2}$  hours a thorough combination will be established, which is perfected by a steam or water-bath.

A clear yellow solid soap having the appearance of a smooth yellow wax-soap is obtained in this manner. After allowing it to cool somewhat it is brought into the frames and perfumed with cassia-oil  $1\frac{1}{2}$  ozs., cinnamon-oil 5 drachms, oil of cloves 2 ozs., oil of lavender  $3\frac{1}{2}$  ozs., tincture of benzoin  $3\frac{1}{2}$  ozs., oil of bergamot  $1\frac{1}{2}$  ozs., oil of winter-green 14 drachms, and tincture of musk  $3\frac{1}{2}$  ozs.

*Mille fleurs soap*.—Cocoanut oil 96 lbs., crude palm oil 4 lbs., caustic soda lye of  $36^{\circ}$  B. 57 lbs.—Orris powder  $17\frac{1}{2}$  ozs., Brilliant Brown 0.3 oz., bergamot oil and geranium oil 2 ozs. each, lavender oil, lemon oil of cloves  $\frac{1}{2}$  oz. each, rosewood oil 0.7 oz., Peruvian balsam  $\frac{1}{2}$  oz., tincture of musk 0.7 oz.

*Musk soap*.—Cocoanut oil 50 lbs., tallow 44 lbs., crude palm oil 6 lbs., caustic soda lye of  $37^{\circ}$  B. 50 lbs., pulverized orange peel 4 lbs.—Cinnabar 7 ozs.—Bergamot oil and lavender oil 7 ozs. each, cassia oil and oil of cloves  $1\frac{1}{2}$  ozs. each, Musk 1:69 drachms macerated in potassium carbonate solution of  $5^{\circ}$  B.

*Rose soap*.—Cocoanut oil 94 lbs., castor oil 6 lbs., caustic soda lye of  $38^{\circ}$  B. 50 lbs.—Oriental Red Rose 0.6 oz.—Palma-rosa oil, lavender oil and citronella oil  $3\frac{1}{2}$  ozs. each.

*Rose soap (white).*—Cocoanut oil 50 lbs., tallow 50 lbs., caustic soda lye of 37° B. 50 lbs.—Geranium oil and bergamot oil 4½ ozs. each, lemon oil 2½ ozs., fine lavender oil 0.17 oz., vetiver oil 0.1 oz.

*Vanilla soap.*—Cocoanut oil 60 lbs., lard 30 lbs., crude palm oil 10 lbs., pulverized cocoa 12 lbs., caustic soda lye of 39° B. 52 lbs.—Vanilla 1½ ozs., balsam of Peru 17½ ozs., oil of lavender 3 ozs., and tincture of musk 1½ ozs.

*Violet soap.*—Melt 63 lbs. of cocoanut oil and 3 lbs. of crude palm oil, cool to 108.5° F., strain, and color with 2½ ozs. of vermillion. Then introduce into the fat through a hair-sieve, with constant stirring, 4 lbs. of pulverized orris-root, 1 lb. of pulverized orange-peel, and ½ lb. of pulverized benzoin. When all the powder is dissolved, saponify the mass with 34 lbs. of soda lye of 38° B. and perfume with lavender oil 2½ ozs., bergamot oil 2½ ozs., oils of cassia and of cloves, each 1½ ozs., and tincture of musk 2 ozs. The soap need not be colored, it naturally being of a beautiful brown color.

*Windsor soap (brown).*—Tallow, cocoanut oil, and soda lye of 37° B., each 50 lbs., potash solution of 15° B. 4 lbs. Perfume with cassia oil 7½ ozs., oils of cumin, cloves and lavender, each 1½ ozs., oil of thyme 1 oz., and neroli petit grain 1¼ ozs., and color with 1½ ozs. of Hessel's Leather Brown dissolved in boiling water.

*Windsor soap (white).*—Saponify 20 lbs. each of tallow and cocoanut oil with 20 lbs of caustic soda lye of 38° B., and perfume with lavender oil 4 ozs., oil of cumin 4½ ozs., citronella oil 14 drachms, fennel oil 11 drachms, and cassia oil 3½ ozs.

*Sand soap.*—Cocoanut oil 100 lbs., caustic soda lye of 38° B. 50 lbs., water 6 lbs., fine silver sand 60 lbs. Cassia oil 6½ ozs., anise oil and mirbane oil 2 ozs. each.

Toilet soaps prepared by the cold process from cocoanut oil with a large percentage of tallow are considerably improved by the addition of 5 to 10 per cent. of *Adeps lanae*, they becoming thereby super-fatted and to make them more brilliant and more like grained toilet soaps, an addition of about 3 to 5 per

cent. of ceresine or Japanese wax is also made. Toilet soaps thus prepared are soft and pliable and have a very pleasant effect upon the skin. They present a very glossy appearance when stamped, and this can be still further improved by polishing with the dry hand. The soaps dissolve with ease, and as they lather more readily than toilet soaps of better quality, are frequently preferred by consumers.

By the cold process super-fatted toilet soaps are made in the same way as cocoanut-oil soaps; the *Adeps lanae*, however, is, as a rule, melted in the fat heated to about 122° F., and then the lye is stirred in at about 89° F. Ceresine or Japanese wax, if used, is added, either by itself or melted with some of the tallow, to the fats heated to about 113° or 118° F., and the lye is then introduced in the usual way. Should the ceresine or Japanese wax, after its incorporation with the oil, partially solidify and commence to separate out on account of the temperature being too low, all of the fat must be warmed again until the ceresine or Japanese wax is redissolved.

A few formulas for toilet soaps with an addition of *Adeps lanae* are here given:

*Fancy soap*.—Cocoanut oil 70 lbs., tallow 24 lbs., castor oil 6 lbs., *Adeps lanae* 4 lbs., caustic soda lye of 38° B. 50 lbs.—Uramine Bright Yellow  $\frac{2}{3}$  oz.—Safrol 5½ ozs., cassia oil 3 ozs., fennel oil and lavender oil each 1½ ozs.

*Princess soap*.—Cocoanut oil 96 lbs., tallow 4 lbs., *Adeps lanae* 4 lbs., caustic soda lye of 38° B. 50 lbs.—Brilliant Rose 0.7 oz.—Bergamot oil and lemon oil each 28 ozs., cedarwood oil and spearmint oil each 1.4 ozs., neroline ¼ oz. dissolved in alcohol, musk tincture ½ oz.

*Peach Blossom soap*.—Cocoanut oil 100 lbs., *Adeps lanae* 6 lbs., caustic soda lye of 38° B. 50 lbs.—Oriental Rose Red 0.17 oz.—Bergamot oil 4½ ozs., palmarosa oil 2½ ozs., oil of cloves, fennel oil, and artificial oil of bitter almonds 0.14 oz. each.

*Spike soap*.—Cocoanut nut oil 90 lbs., tallow 10 lbs., *Adeps lanae* 4 lbs., caustic soda lye of 38° B. 50 lbs.—Sultana Yellow

0 1 oz.—Lavender oil 5½ ozs., spike oil 2¾ ozs., geranium oil, patchouli oil, and palma-rosa oil 1¼ ozs. each.

*Super-fatted toilet soap.*—Cocoanut oil 100 lbs., *Adeps lanae* 5 lbs., caustic soda lye of 38° B. 50 lbs.—Ginger grass oil 7½ ozs., terpineol and bergamot oil 1.41 ozs. each, oil of cloves 1 oz., patchouli oil ½ oz.

*Super-fatted toilet soap (yellow).*—Cocoanut oil 84 lbs., tallow 16 lbs., ceresine 3 lbs., *Adeps lanae* 4 lbs., caustic soda lye of 38° B. 50 lbs.—Sultana Yellow 1.69 drachms.—Lemon grass oil 6½ ozs., lavender oil, cassia oil, citronella oil, each 3½ ozs., tincture of musk ½ oz.

*Super-fatted toilet soap (green).*—Cocoanut oil 100 lbs., *Adeps lanae* 5 lbs., caustic soda of 38° B. 50 lbs.—Leaf Green M ½ oz., Uramine Bright Yellow 0.12 oz.—Bergamot oil and citronella oil each 3½ ozs., patchouli oil 2.6 ozs., artificial oil of bitter almonds ½ oz.

*Super-fatted toilet soap (brown).*—Cocoanut oil 105 lbs., *Adeps lanae* 5 lbs., caustic soda lye of 38° B. 50 lbs.—Brilliant Brown ½ oz., Sultana Yellow 2½ drachms—Bergamot oil 4½ ozs., cassia oil 4½ ozs., lemon oil and safrol each 2 ozs.

*Violet soap.*—Cocoanut oil 96 lbs., tallow 4 lbs., *Adeps lanae* 2 lbs., caustic soda lye of 38° B. 50 lbs.—Brilliant Brown ½ oz., cinnabar 2.4 ozs.—Lavender oil 5½ ozs., Peruvian balsam 3.8 ozs., cassia oil 1½ ozs.

*Toilet soaps by remelting.*—The preparation of these soaps forms a special branch of industry, they being manufactured from soaps of various kinds already made—so-called stock soaps. The latter are remelted and then mixed with perfume and coloring matter. Tallow, cocoanut oil, palm oil and castor oil are used for the preparation of the stock soaps, and they should be carefully made, be as free from excess of caustic soda as possible, and contain but little or no salt. High-grade perfumes should not be used for remelted soaps, because the fine, delicate scent would to a great extent be evaporated whilst being incorporated with the hot soap.

The process of preparing the soaps is as follows:

The stock soap is first in the usual way cut into thin slabs by a wire or converted into shavings. For remelting, kettles of various sizes, holding from 50 to 300 pounds, are used. A very convenient form of remelter consists of a jacketed pan, provided with a stirrer so that the soaps can be intimately mixed.

Fig. 37 shows an improved kettle for rendering and refining toilet soap. It is a seamless, steam-jacketed kettle, provided with an agitator so constructed that it can be easily removed from the kettle and swung out of the way when no agitator is required, or for cleaning the machine.

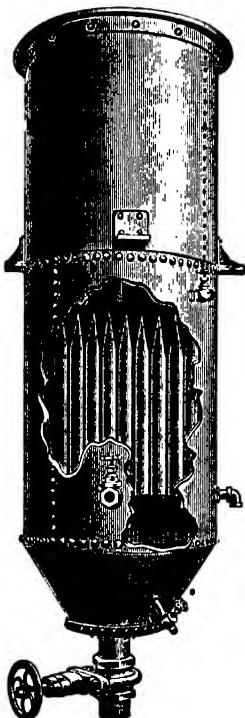
FIG. 37.



*A* is an upright provided with a rack screwed into a bracket cast on the kettle. A pinion, operated by a hand-wheel, *E*, engages with this rack, and thus the agitator can readily be raised out of the kettle. On reaching the top it can be swung to one side out of the way, and the kettle can be used for boiling and all purposes to which a steam-jacket kettle can be put. The agitator consists of a conveyor-screw, *D*, surrounded by a cylindrical casing, *C*. By loosening a set-screw, the conveyor-screw, *D*, can be withdrawn, and the whole machine can be cleaned very easily. It can be run by hand or power.

As the agitator can so readily be taken apart and thoroughly cleansed, and the machine has all the needed facilities for heating or cooling the mass by running cold water through the jacket, the kettle is very useful to soapmakers who make fancy toilet soaps.

FIG. 38.



Some forms of remelter have a number of upright steam pipes or tubes fixed in the machine. The heat is thus brought very quickly to the very centre of the soap mass, which is advisable, as soap is a bad conductor of heat, but it also saves time in effecting the operation.

The "Acme" remelter, Fig. 38, manufactured by the

Houchin-Aiken Co., Brooklyn, N. Y., is of improved mechanical construction. It consists of a cylindrical steel shell open at the top, with a conical cast-iron bottom having an outlet in the center with a quick-acting gate valve. The remelting coil is supported just above the conical bottom, and consists of a series of vertical pipes screwed into a cast-iron header. An open steam coil is placed near the bottom of the remelting coil to permit of the addition of live steam to the remelted soap when desired. The conical bottom, extending below the remelting coil, forms a reservoir for melted soap, a closed steam coil being provided at this part to keep the melted soap from cooling.

Remelters should be set up sufficiently elevated so that a soap frame can be conveniently placed underneath. This may be done either by placing the remelter on a platform of desired height, if the height of the room permits, or by suspending it through the floor above the one on which the soap is framed. If, however, all the apparatus has of necessity to be placed on a level, the soap has to be conveyed from one machine to the other, which in small factories is sometimes done by hand labor, ladles or scoops being used for the purpose. This process is, however, time-consuming and expensive and the better method is to use a pump. A pump largely used for this purpose is of the type known as the rotary pump (see Fig. 14, p. 189).

Below a few formulas for toilet soaps by remelting are given. They may serve as a guide to the soapmaker, who can make up a formula of his own, using any particular perfume he may wish and any color he likes.

*Almond soap.*—White tallow grained soap 100 lbs., olive oil soap and cocoanut oil soap each 14 lbs., oil of bitter almonds 26 ozs., oils of clove and caraway each 8 ozs., or in place of the latter, rose oil  $\frac{1}{2}$  oz.

It is best first to melt one-half of the tallow-soap, then the other two soaps, and when they are melted add the other half of the tallow-soap, and finally the perfume. By substituting nitro-

benzole (essence of mirbane) for all or a portion of the oil of bitter almonds, a cheaper soap of not as fine a quality is obtained.

*Camphor soap.*—Tallow grained soap 100 lbs., oil of rosemary  $4\frac{1}{2}$  lbs., camphor  $4\frac{1}{2}$  lbs.

Reduce the camphor to powder by triturating it with some almond oil, then sift it. When the soap is ready to be brought into the frame add the camphor and rosemary oil, with vigorous crutching.

*Windsor soap (old brown).*—This popular soap is made in the following manner: Convert 50 lbs. each of palm soap and half-palm soap into shavings and spread upon sheets of strong paper to dry; when dry melt on a water-bath with a small portion of an aromatic water, and when it is hard enough cut into shavings as before, drying it again and remelting and adding sugar color. After the third operation add the following perfume for the 100 pounds: Oil of bergamot 4 ozs., oils of caraway and cassia, each 2 ozs., lavender 8 ozs., clove and petit grain, each 1 oz.

Brown Windsor soap owes its fine emollient properties to the amount of labor employed in its manufacture, for it is needless to say that the more the soap is worked and handled and melted and remelted the better it becomes.

*Heliotrope soap.*—Tallow soap 20 lbs., oil of almonds  $\frac{1}{2}$  oz., neroli oil 1 oz., benzoin  $1\frac{1}{2}$  ozs.

*Bouquet soap.*—Tallow soap 80 lbs., cocoanut oil 20 lbs., bergamot oil 3 ozs., rose geranium oil 2 ozs., caraway oil and citronella oil each  $1\frac{1}{2}$  ozs., lavender oil 1 oz.

*Milled toilet soaps.*—These are the best and finest toilet soaps, and it is of great importance that the stock soaps used in their preparation should be made from fresh and pure materials and contain no incompletely saponified fat which would exert an injurious effect upon their durability. They should be perfectly neutral, *i. e.* contain no free alkali which attacks the skin and causes to a delicate and sensitive one an unpleasant burning sensation. An important requisite is, however, that the scents

imparted to the soaps by the addition of perfumes shall be full, pure, and without unpleasant accompanying odors. They should last as long as possible, in many cases for a year or so, which will not be the case if the stock becomes rancid in the slightest degree. The aim of the manufacturer should therefore be to employ a pure neutral soap, possessing no smell of its own for working up into milled soap, and it is the office of the soapboiler who prepares the stock soap to see to it that these conditions are fulfilled. From this it will be seen that the preparation of stock soaps requires more care and greater attention than that of ordinary soaps. The necessity of being free from all smell of their own applies to all stock soaps except palm oil soap, which always retains the odor of palm oil, this odor being often desired as it is suitable for strengthening many perfumes.

The selection of animal as well as vegetable fats and oils suitable for the preparation of stock soaps depends on circumstances. In temperate and northern latitudes the beef tallow produced in these localities is for reasons of convenience and economy employed and is very suitable for the purpose, while in southern countries the less valuable grades of olive oil are employed. Lard is also a valuable material for stock soaps, provided it is not rancid. It is frequently brought into commerce apparently odorless, but on saponification acquires an unpleasant smell, and in all cases of unknown origin it is recommended to guard against loss by first saponifying a sample and examining the resulting product for smell. A good grade of freshly-expressed peanut oil is also an excellent addition in making stock soaps, though more than 25 per cent. of it is not advisable as the use of too much of it has a tendency to impair the hardness of the soap.

Drying oils such as cottonseed oil, castor oil etc., should not be employed in the preparation of stock soaps, as they will sooner or later become rancid. This applies also to mutton and goat tallows, though they are very suitable for household soaps.

In regard to the alkalies used for the saponification of the fats, it may be said that caustic soda could formerly not be procured of such purity or at as low a price as at present and lyes of 10° to 12° B., prepared by treating sodium carbonate with burnt lime were used for boiling stock soaps. However, lyes containing a large amount of carbonate yield soaps of little durability, and as lyes of the above mentioned strength were the strongest that could be used, the soap had to be boiled several times with fresh lye and, after salting out, the lye previously used had to be drawn off. The soap was finally once more boiled up with fresh lye and brine, the spent lye again removed and then the whole batch again treated with a weak brine until a thin liquid soap resulted which was allowed to stand for some time to settle, and then brought into the frame. With the caustic soda of great purity now obtainable, such a tedious process is no longer necessary, though to obtain the required durability of the product, it is always best to treat the soap at least three times with fresh lye, because by the repeated separation of the spent lye the greatest part of the mechanical impurities contained in the fat, lye, and salt are removed from the soap, and the consecutive treatments with fresh lye completely saponify every particle of fat.

The quantity of caustic soda required for saponification can be readily calculated when it is borne in mind that 220 lbs. of it produce 660 lbs. of lye of from 38° to 40° B., and that 220 lbs. of this lye are sufficient for the saponification of 440 lbs. of fat to obtain a neutral soap. Before proceeding with the boiling it is advisable to calculate the quantity of caustic soda required for a fixed amount of fat. Supposing 6600 lbs. of fat are to be worked up into stock soap, 1100 lbs. of caustic soda would be required for saponification, because, as 2.2 lbs. are sufficient to saponify 13.2 lbs. of fat,  $1100 \times 6 = 6600$  will be necessary for the above-mentioned quantity of fat. The 1100 lbs. of caustic soda are to be dissolved in water in a holder by suspending the drum with a pulley as previously described.

In the boiling of soaps it is not always necessary to invariably employ a fixed strength of lye; it depends upon whether direct fire or steam is used, and in the latter case, whether the steam is wet or dry according to pressure and the distance it has to be conveyed. Stock soaps should, however, as a rule be boiled with not too strong a lye, because saponification is then more thorough and the durability of the soap is consequently improved. A convenient way is to make the lye up about  $25^{\circ}$  to  $26^{\circ}$  B., and reduce one-third of this to from 12 to  $15^{\circ}$  B. according to whether direct fire or steam is used. This reduced lye is placed in a special reservoir. Another third is reduced to  $18^{\circ}$  or  $20^{\circ}$  B. according to the same circumstances. Bring all of the fat, with the exception of the cocoanut oil, into the kettle and commence saponification with the weakest lye, being sure that all the lye enters into combination before adding fresh lye. The lye should always be near at hand so that it can be immediately added, when required, to prevent the soap forming irregularly. When the weakest lye has been used up, the second is added and the boiling is continued until free from caustic "touch." The soap is then salted out until a clear lye forms underneath which, after standing for two hours, or still better the next day, is removed by means of a pump, or according to circumstances syphoned off into a second kettle. A further quantity of brine of  $10^{\circ}$  to  $12^{\circ}$  B. together with about 220 lbs. of the remaining strong lye, is run into the kettle and the soap boiled with it for some hours. This process is required for the durability of the soap and its necessity is clearly indicated by the color of the spent lye. With a pure soap this lye should be as clear as water; a brown color indicates that the soap still contains impurities which must be removed by treatment with weak salt water. After the spent lye has been repeatedly separated, the rest of the soda lye is finally run into the kettle; the soap will now be strong, but that does not matter. It is now boiled for two hours to ensure complete saponification. The cocoanut oil is then added and this

neutralizes the excess of caustic lye, when the soap should boil light and liquid, and is now ready for fitting. The soap should not produce a biting sensation upon the tongue, as with such an excess of alkali it would not be sufficiently mild, would sweat if kept in stock, and probably would also change in color. To fit a stock soap properly and with safety it should be tested with phenolphthalein solution as previously described.

The soap should now show all the characteristics of a well made product, and above all it should be well separated and of good consistency, otherwise it must be treated with brine of 2 to 3° B. until it is sufficiently thin. It is then ready to be further worked up in the frame.

Below a few combinations of fats for stock soaps are given:

*The stock soaps* for white and colored soaps are generally prepared from 9 parts of fresh tallow free from odor and 1 part of cocoanut oil, the addition of the latter securing a more abundant lather. Frequently more cocoanut oil is used, but the above composition yields the most durable soaps. Other compositions are as follows:

I. Fresh tallow and cottonseed oil each 50 parts, cocoanut oil 10 to 25 parts.

II. Fresh tallow 75 parts, peanut oil 25 parts, cocoanut oil 10 to 25 parts.

III. Fresh tallow and lard, each 50 parts, cocoanut oil 10 to 25 parts.

*Stock soaps for cream, yellow, and brown soaps.*—I. Bleached palm oil 100 parts, cocoanut oil 10 to 25 parts.

II. Bleached palm oil 50 parts, tallow or lard 25 parts, peanut oil 25 parts, cocoanut oil 10 to 25 parts.

The stock soaps may be made up in any quantity desired and from them nearly all kinds of toilet soaps can be made by mixing different kinds in suitable proportions, according to demand and ruling prices.

Stock soaps after being taken from the frames should be cut into bars as soon as possible and then dried in the air or dry-

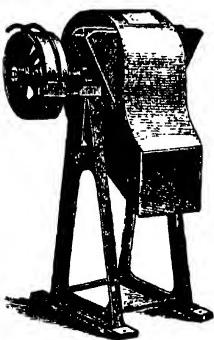
ing room to reduce them as rapidly as can be done to the necessary degree of dryness for further operations.

The first operation is to "strip" the stock soap, *i. e.*, to cut it up into strips or shavings. This may be done either by hand, which is rather a primitive way, though suitable and economical for small lots of soap, or by a machine called a chipper or stripper.

The tools required for working by hand consist of an ordinary carpenter's plane and a good marble mortar and pock-wood pestle. Each end of the plane should be provided with a contrivance, so that when placed over the mortar it remains firm and is not easily moved by the parallel pressure of the soap against the projecting blade.

The operation commences by weighing determined quantities of the soap that is to be cut up and perfumed. The plane

FIG. 39.



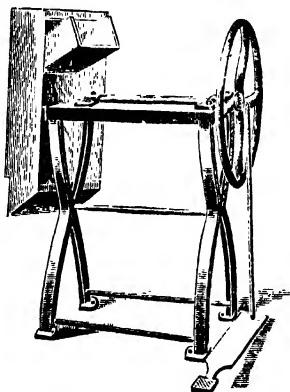
is then laid upside down across the mortar and the bar of soap pushed across the plane until it is reduced to fine shavings. Soap received from the soap-maker is generally in proper condition for thus working; but if it has been in stock any time it is apt to become too hard, and after having been converted into shavings must be sprinkled with some distilled water and allowed to stand 15 to 24 hours to give the shavings time to absorb the water before the perfume is added.

After determining the size the cakes of soap are to be, what they are to sell for, and what they are to cost, the maker can measure out his perfume.

The soap being in proper condition in regard to moisture, etc., is now to have the perfume well stirred into it. This is done by working it thoroughly with the pestle for a few hours, when the soap is generally expected to be free from streaks and of uniform consistency.

The soap thus perfumed is then weighed out in quantities as required for the tablets and moulded by the hand into egg-shaped masses, which are laid separately in rows on a sheet of white paper, and allowed to dry for a day or two to fit them for the press. It is usual before placing the cakes in the press to dust them over with a little starch-powder, or very slightly to

FIG. 40.



oil the mould, to prevent the soap from adhering to the letters or embossed work of the mould.

For the preparation of large quantities of toilet soap it is more convenient and economical to use machinery.

The stock soap is cut into shavings by a machine called a "stripper" and driven either by hand or power.

There are several different kinds of this machine, though the essential parts of all of them are one or two revolving disks provided with four to six knives and a hopper to contain the bars. Fig. 39 shows such a machine with two disks. Opposite the disk is a hopper or cylinder in which the bars of stock soap to be stripped are placed. By pressing the bars against the disks, they are cut into shavings, which fall through slits in the disks into a receptacle.

Fig. 40 shows a 20-inch disk-chipper manufactured by Houchin-Aiken Co., Brooklyn, N. Y. It is fitted with six knives. The machine will take bars 4 x 4 inches cross-sections. It is furnished to run by foot-power, or, if desired, can be equipped with pulleys to run by power.

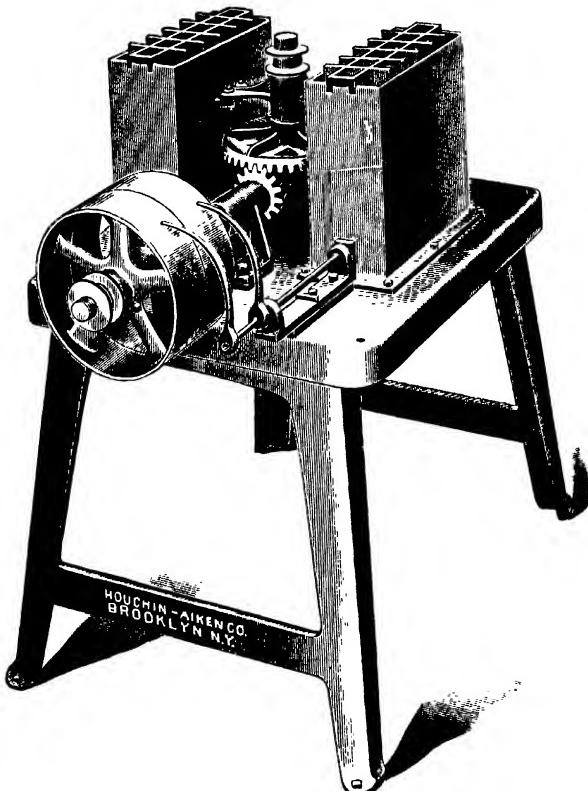
Fig. 41 shows a 30-inch horizontal disc-chipper manufactured by the above-mentioned firm. It has a 30-inch balanced disc, fitted with 12 knives 2 x 9 inches. The disc is supported and driven from above through cut-gears. The knives can be adjusted to give any thickness of chip desired. The machine will cut two slabs of soap at once as large as 5 inches thick by 15 inches wide, or, by means of a simple device, 36 small bars may be fed to the machine at once. The chipped soap falls vertically from the machine.

The shavings, if necessary, are dried by spreading them out in trays. They should not be piled up too high so as to allow the drying air to pass freely through them. When dry the required quantity of shavings is weighed out and brought into the mixing box lined with zinc or lead, which is for greater convenience mounted on wheels so that it can be readily moved about from place to place. The proper proportions of essential oils \* and coloring matter (except when the soap is required to be white) are then added and the whole is intimately mixed together with a wooden stirrer. When working on a larger scale this is done in an automatic mixer, an im-

\* Essential Oils and Other Materials used for the perfuming of soaps are described in Chapter XVIII.

proved device for this purpose being a mechanical amalgamator which breaks up and amalgamates the chips, perfume and coloring matter. The shavings are next conveyed to the *Soap-mill* to be blended into a thick homogeneous paste.

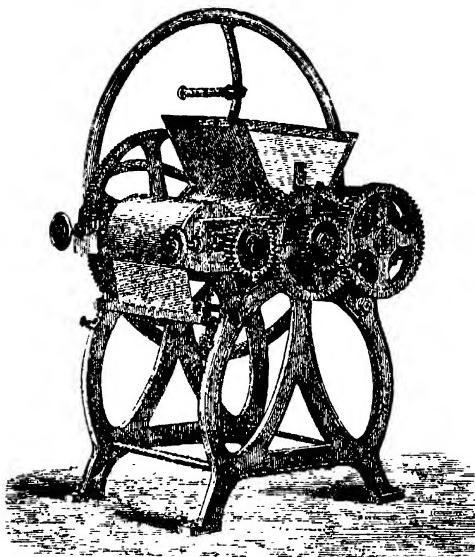
FIG. 41.



The mill is similar in construction to a cocoa-mill, and consists essentially of three or four contiguous rollers by whose action the shavings, color, and perfume are intimately united. The size of the mill, and, whether it is to be worked by hand

or steam, depends on the size of the establishment. Fig. 42, shows a mill with three rolls to be worked by hand. The rolls are of best syenitic granite finely polished. The perfumed and colored shavings are brought into the hopper, and after passing through between the first two rolls fall upon the third. A scraper pressing against the third roll scrapes off the soap which falls into a receptacle. The milled mass must be re-

FIG. 42.

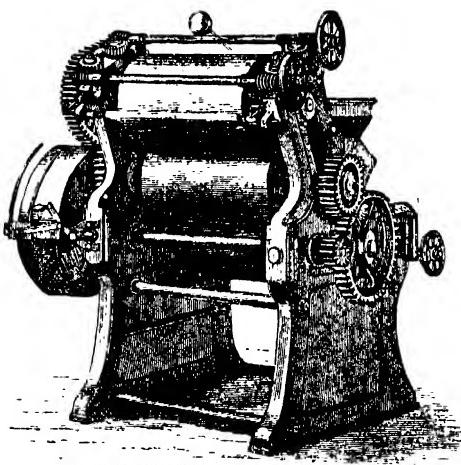


turned to the hopper and again passed through the rolls until it is perfectly homogeneous.

With a machine with four rolls, Fig. 43, this repeated passing of the milled mass through the rolls is not required, since the operation is effected by the fourth roll. Besides, in large factories where the work is carried on continuously, two or three machines may be placed alongside each other and the thorough mixing of the mass effected by passing it successively through the machines.

Fig. 44 shows a soap mill constructed upon different patterns and principles. The bed is of box-section extending to the ground and on an incline, and with long bearings for the stone rollers, which are made from the best Quincy granite, a material well adapted for the purpose on account of its hardness, the surface finishing true and smooth, yet porous, so as to grind the soap quickly and perfectly. The shafts, which extend entirely through the rollers, are of steel and very heavy,

FIG. 43.

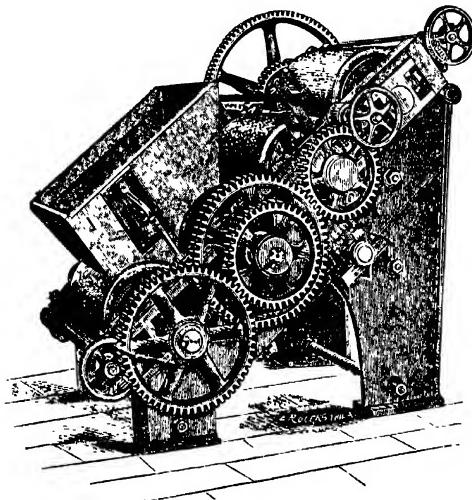


so as to insure against all possibility of springing, and are fastened in the stone rollers with a special device which renders it impossible for them to become loose on the stone. The gearing and all the wheels are either machine moulded or machine cut. The pulleys are of large diameter and breath and capable of transmitting ample power to the working parts of the machine. The stone rollers are automatically adjusted by a wheel and worm, which motion is positive, insuring the whole length of the rollers working accurately together, by which

the soap is ground thoroughly and rapidly and with the least possible wear on the machine. The mill has a capacity of 250 pounds per hour; power required, 12 horse-power.

The homogeneous mass, free from grains and streaks, is removed from the last roller in the form of long bands by means of knives fixed to the machine and falls into the mixing box placed below in which it is conveyed to the plodder. The object of this machine is to compress the colored and perfumed

FIG. 44.

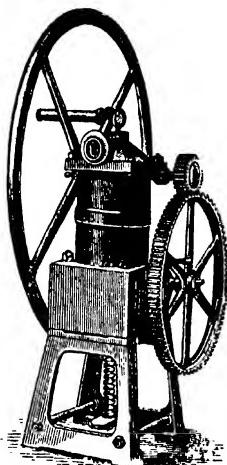


soap after milling into a solid compact mass. It then ejects it in the form of a bar which is then divided up into cakes of the desired size. Machines of various construction are employed for this purpose.

Fig. 45 shows a plodder worked by hand. It consists of a cylinder supported vertically upon a stand and provided at the delivery end with a changeable mouth-piece. A plunger exactly fitting the cylinder is moved up and down by hand or machine power. The cylinder is filled to the top with the

soap, the plunger being raised so that this can be conveniently done. On setting the machine in motion the plunger descends upon the soap and compresses it in the cylinder, the opening by which the soap eventually leaves the machine having previously been closed. When the soap is sufficiently compressed the plunger is again raised and the empty space left by the compression of the soap filled with soap which in its turn is compressed, the operation being thus continued un-

FIG. 45.

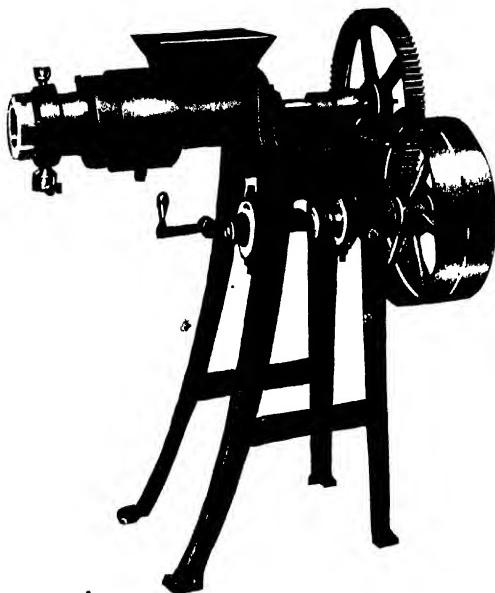


til the cylinder is filled to the top with compressed soap. The plunger is now left some minutes under pressure in the cylinder. It is then released, the delivery end opened and a mouth-piece of the shape the cake of soap is to have inserted. The plunger is then allowed to work with full force upon the soap, forcing it in the form of a cake of any shape desired, according to the mouth-piece used, through the opening.

However, as the operation with such a machine is quite slow, it is now largely superseded by the *screw plodder*. This machine consists essentially of a screw enclosed in a cylinder, by

which the soap is gradually driven forward and at the same time subjected to a considerable degree of compression, it being thus consolidated and made firm. The cylinder of the machine is at first double, thus forming a jacket in which cold water circulates for the purpose of keeping the soap cold, the friction and pressure causing a great deal of heat to be generated ; this heat might, if allowed to accumulate, cause loss of perfume

FIG. 46.

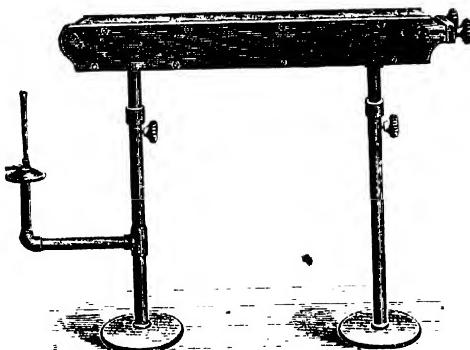


and color in the soap. The mouth-piece of the machine is kept hot by means of a gas-jet, usually supplied with each machine, the purpose of heating being to impart a gloss and finish to the soap. The soap passes very rapidly through the machine and leaves the latter in a continuous bar, sections of which are removed as fast as ejected and cut into individual cakes.

Fig. 46 shows a type of plodder manufactured by Houchin-Aiken Co., Brooklyn. It has a 4-inch screw of special construction, which gives it a rapid feed. The cylinder is water-jacketed. The front nose is screwed to the cylinder and in front has a jacket for heating. The cylinder, screw and front nose are enameled, except in the working parts. The gears are of cast-iron, machine cut.

Fig. 47 shows a roller-board. This device is placed in front of the plodder to receive the bar of soap as it comes from the

FIG. 47.



plodder. It will not mar the soap. It has a stand for a gas-burner for heating the front nose of the plodder.

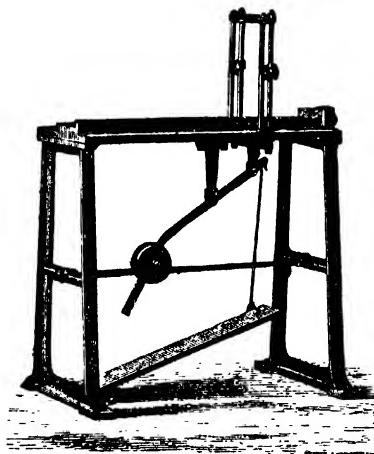
*Cake cutters.*—When the plodder is not provided with a cutting apparatus a special machine for the purpose is required. These machines are of various sizes, and being entirely constructed of iron, are very durable. The working table (Fig. 48) is smooth planed. The horizontal wire moves up and down upon two small vertical columns by treading upon the treadle under the table, the counter-weight again raising the wire. A shiftable back-square determines the length of the cakes. The machine being worked by a treadle, both hands are left free; the left pushing the bar forward, while the right removes the

cakes. This machine requires but little force, so that it can be run by a girl.

Before pressing, the cakes must be heated to facilitate the work and obtain a fine impression, and after pressing the cakes have to be dried. This is effected in a drying-room heated either by a stove or hot air.

*Soap-presses.*—The cakes being sufficiently dry, it is frequently necessary to give them a finish, which is often done in a hand-press in a plain mould; but occasionally a mould-box

FIG. 48.



with hinged sides is employed, with a screw-press such as shown in Fig. 49. For large tables a foot-power press is desirable. Fig. 50 represents a foot-power press. It is worked by a treadle, leaving the hands free to handle the soap. It has adjustable balances to vary the blow. It works easily and can be run by a boy. It is adapted from the smallest size toilet soap to a two-pound laundry soap.

The soap-stamping press, shown together with box and dies in Fig. 51, is manufactured by Houchin-Aiken Co., of Brook-

lyn. This press is adapted for stamping any sized cake of soap, is simple in construction, and adjusted to any sized dies. It has a vertical treadle motion, is easily worked, and gives a direct blow to the soap. The box and dies for stamping soap are made of hard brass and consist of an upper and lower die

FIG. 49.

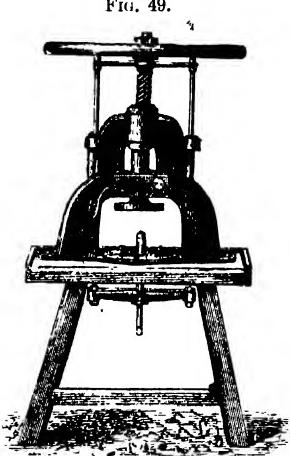
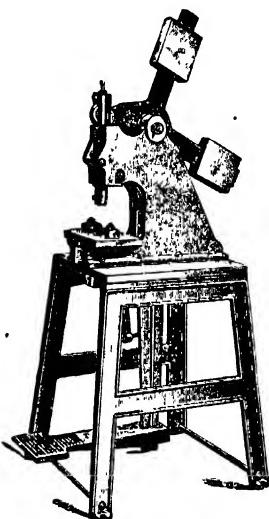


FIG. 50.



fitted to a box, which is clamped to the bed of the press, with the bottom die in the box, the top die being fastened to the plunger. The dies can be engraved of any desired size and shape to suit, or can be made with interchangeable plates for the engraving. By this means different brands of soap can be pressed with the same die by only changing the plate.

Fig. 52 shows a steam soap-press. It has a single-acting steam cylinder placed underneath the bed in such a position that its piston, by means of a roller attached to the end of the piston-rod, acts upon a cam surface of the swing or pendulum-lever, as indicated at *B*. A hook, *A*, attached to the piston-

rod engages with a stud on the swing or pendulum-lever, and prevents the latter from recoiling after having returned from giving the blow, as it cannot fly back without pulling out the piston. Thus the unpleasant and dangerous vibration of the upper die-block is prevented. The steam-supply pipe enters a governor or regulator which can be set by hand-wheel, *E*, so

FIG. 51

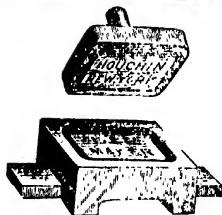
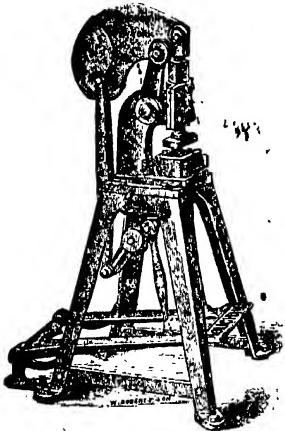
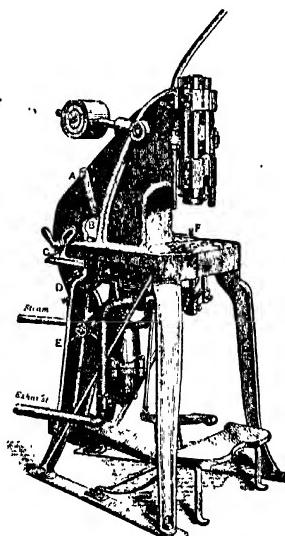


FIG. 52.



that the press gives a blow of required force. When this has once been set, the press cannot give a stronger blow than that for which it is set, no matter how much steam pressure the boiler may supply. To the right of this governor, *E*, is shown a balanced valve steam-trap which drains off all condensed water, and insures the admission of dry steam only to the

cylinder, no matter how far the press may be from the boiler. The admission of steam is controlled by a foot-treadle, shown at the right of the cut, the heel of the foot resting on the foot-rest. The handle, *F*, serves to control the exhaust in such a manner that the pendulum-lever returns with just enough force to eject the pressed soap, and no more. This ejection of the soap is accomplished by a cam, *C*, which is pivoted at one end to the pendulum-lever, and clamped to the latter by a jam-nut and arcs. Against this cam works, by means of a roller, a lever which with its other end actuates the center lifting bolt. By unclamping this cam, shifting it up or down, and reclamping, the height to which the soap is lifted is regulated. This arrangement lifts the soap so gradually that there is no danger of throwing the cake of soap out against the upper die-block, and defacing the impression, no matter how fast the press is worked.

By throwing back hook *A*, and raising the foot-rest, the press is at once transformed into an ordinary foot-press. This is a great convenience in setting the die, as well as furnishing means of working when there is no steam.

*Finishing and polishing the soap-cakes.*—The cakes of soap, after they are pressed, stamped, and apparently ready for market, are often dried before being packed. In this drying they may lose some of their lustre. The remedy formerly used for this was to scrape the cakes off, and rub them with a woolen cloth dipped in strong alcohol.

This somewhat troublesome process has been superseded, according to Dupuis, by the method of exposing the soap before or after drying to a current of steam. The steam can be perfumed with any fragrant odor by passing it, before reaching the soap, through a cloth impregnated with fragrant material. The steam causes at once a change upon the surface of the bars or cakes of soap, and forms, according to the fats used in their preparation, either a super-palmitate or super-stearic palmitin soda combination. If this operation is carefully done it closes up all the pores and uneven spots and, when dry,

forms a very lustrous coating, which does not suffer, even under the moulding-press. No other method of polishing will give such a beautiful, even, and lustrous coating as that caused by steaming. Further advantages of this mode of operating are economy in time, manual labor, and prevention of all loss. It will especially preserve the soaps in damp storehouses, on sea voyages, and in the show-windows of stores, where they are exposed to the rays of the sun.

*Coloring toilet soaps.*—Whilst in former times ochres, chromes, and metallic oxides of iron, such as siennas, umbers, etc., were exclusively used for coloring toilet soaps, the object is now largely attained by means of aniline colors. They, to be sure, have the drawback that the soaps when exposed to sunlight or diffused daylight are apt to lose or change their color. The chief colors used for toilet soaps are red including rose, and yellow, as well as the shades lying between them, such as orange, chamois, salmon-red, and others. All these shades can be produced by the mixing of two colors, rhodamin and fluorescein.

Rhodamine represents a red-brown powder and dissolves with ease in 100 times its weight of pure water. Concentrated solutions may also be prepared by adding about 10 per cent. of alcohol to the water. One grammie of rhodamine is used for 220 lbs. of soap. With cocoanut-oil soap it gives a fine bluish-red tint. For milled soaps, which are generally not so deeply colored as cocoanut oil soaps,  $\frac{1}{2}$  grammie suffices for 220 lbs.

Fluorescein dissolves readily in 10 parts of water, the solution possessing a strong fluorescence which is noticeable in transparent soaps colored with it, but does not show in colored opaque soaps. For imparting a full citron color to 220 lbs. of soap, 5 grammes of fluorescein are required.

Two solutions are now prepared, one of them containing 100 grammes rhodamine in 20 liters of water, and the other 500 grammes fluorescein in 20 liters of water, therefore for coloring 220 lbs. of soap 100 cubic centimeters will be used.

When shades lying between the yellow and the red are required, the two solutions are mixed until the desired combination is obtained. A salmon-red, for instance, can be produced with 80 ccm. of the red and 20 ccm. of the yellow solution, a bright orange with 50 ccm. of each; a household soap yellow with 90 ccm. of the yellow solution and 10 ccm. of the red. These solutions can be kept in stock and if by any reaction between the two dyestuffs a precipitate is produced, add just enough caustic soda to redissolve it.

Metanil yellow is very suitable for coloring toilet soaps. It dissolves with somewhat greater difficulty than the above-mentioned coloring matters and distilled water is preferably used for dissolving, as with lime salts in the water it forms an insoluble precipitate. One-half to one grammé of it is required for 220 lbs. of soap. In cocoanut-oil soaps, a similar quantity produces a full, bright yellow.

Chinoline yellow, auramine, and mandarin have also been recommended for coloring toilet-soaps yellow.

There is but one single green dyestuff which is a self-color and suitable for soaps. This is naphthol green, known as soap green, which is easily soluble in 10 parts of water. A solution containing 400 grammes naphthol green and 100 grammes of fluorescein in 10 liters of water is recommended. For coloring 220 lbs. of soap about 100 ccm. of this solution are to be used. Naphthol green being sensitive to an excess of alkali, especially when hot, and being partly decomposed thereby, should be added to the melted soap after it is completely saponified.

There is no brown dyestuff known which is a self-color and suitable for coloring soap. The brown coloring matters are composed either of mineral colors such as umber and sienna, or mixtures of both, which are added to the fat during saponification, or they are mixtures of orange or yellowish-red colors with blue dyestuffs. Most brown colors change their shade when stirred up with lye. With aniline colors quite fair shades of brown can be obtained as follows; Naphthol orange

or mandarin 40 grammes, fast blue 10 grammes, soluble in water; or naphthol orange 20 grammes, methyl blue 3 grammes. The mixtures are dissolved with the addition of a little caustic soda in about 1 liter of hot water.

For heliotrope soap which should have a bright lilac color a mixture of 200 grammes of ultramarine and 100 ccm. of rhodamine solution may be used for 220 lbs. of soap.

A few formulas for milled soaps are given below:—

*Elder-flower soap.*—Half-palm soap 200 pounds, dextrine 6 pounds. *Perfume:* oil of bergamot 1 pound, oil of lavender 2 ounces, oil of thyme 4 ounces, oil of cloves 2 ounces and oil of cassia and almonds each 1 ounce, *Color:* light green.

*Another formula* is as follows: Tallow stock soap 100 lbs. *Color:* 3½ ozs. light cinnabar, 1.4 ozs. ultramarine. *Perfume:* 14 ozs. terpineol, 14 ozs. bergamot oil, ¾ oz. artificial oil of bitter almonds, ½ oz. vaniliane, 6 ozs. artificial ylang-ylang oil, ½ oz. aubepine, 5½ ozs. geranium oil, 0.14 oz. artificial musk.

*Lemon-soap.*—White soap 100 pounds, starch 4 pounds. *Perfume:* oil of lemon 8 ounces, oils of bergamot and lemon grass each 4 ounces, oil of cloves 2 ounces. *Color:* yellow with cadmium yellow.

*Orange-soap.*—White soap 100 pounds, starch 4 pounds. *Perfume:* oil of orange-peel 16 ounces, oil of cinnamon 1 ounce and oil of thyme 4 ounces. *Color:* dark yellow with naphththylene yellow.

*Patchouli-soap.*—White stock-soap 50 pounds, potato-flour 10 pounds. *Color:* 1½ ounces of Cassel brown. *Perfume:* geranium-oil 3½ ounces, oil of patchouli 7 ounces.

*Another formula* is as follows: Palm oil stock soap 100 lbs. *Color:* 7 ozs. May green. *Perfume:* 29 ozs. lavender oil, 3 ozs. patchouli oil, 0.8 oz. oil of vetiver.

*Heliotrope soap.*—White stock soap 100 lbs. *Color:* 1.4 ozs. Lavender blue, 1.4 ozs. light cinnabar. *Perfume:* 26 ozs. heliotropine, 1.4 ozs. vanilline, ½ oz. artificial oil of bitter almonds, ½ oz. eugenol, 1.12 drachms artificial musk.

*Frangipanni soap.*—Palm soap 30 lbs., white soap 20 lbs.

dextrine 2 lbs. *Perfume*: Oil of bergamot 4 ozs., oils of neroli and santal each 2 ozs., tinctures of vanilla and civet each 8 ozs. Color light brown with tincture of catechu.

*Cold-cream soap*.—White soap 30 lbs., spermaceti soap 20 lbs., oil of almond  $\frac{1}{2}$  lb., caustic potash of 6° B. 1 lb., gum tragacanth 2 ozs.

Strip up the two soaps, place them in the hopper of the mill, dissolve the gum by previous soaking in a little water, mix with the oil and lye to a uniform consistency, then stir into the soap and grind in the mill until thoroughly combined. Care should be taken to have the mass as white as possible. *Perfume* the above with oil of bergamot 5 ozs., oils of cloves and nutmeg each 1 oz., oil of thyme 2 ozs., oil of bitter almonds 1 oz.

*Savon de riz*.—54 lbs. of wax soap and 8 lbs. of starch. *Perfume*: Oil of geranium 1 oz., oil of orange (Portugal)  $1\frac{1}{2}$  ozs., oil of bergamot  $1\frac{1}{2}$  ozs., oil of mirbane 1 oz., tincture of benzoin  $\frac{1}{2}$  oz. The soap remains white, or is colored red with  $2\frac{1}{2}$  ozs. of vermillion.

*Savon au bouquet*.—White stock soap 30 lbs. *Perfume*: Bergamot oil  $1\frac{1}{2}$  ozs., oil of sassafras  $\frac{1}{2}$  oz., oil of thyme 6 drachms, oils of lavender and cloves each 5 drachms, oil of geranium 2 drachms. *Color*: Yellow, brown, red or green.

*Herb soap (Dr. Borchardt's)*.—White stock soap and olive oil soap each 60 lbs., starch 3 lbs. *Perfume*: Oils of cassia and lavender each 1 lb., and oil of bergamot 2 lbs.

*Lily soap*.—Wax soap 60 lbs., starch 6 lbs. *Perfume*: Oil of bergamot 5 ozs., oil of geranium 2 ozs., oil of cassia  $\frac{1}{2}$  oz., oil of santal 3 drachms, oil of cedar, tincture of musk, and tincture of tonka-bean each 1 oz., tincture of storax 3 ozs.

*Canaga soap*.—Palm oil stock soap 100 lbs. *Perfume*:  $21\frac{1}{2}$  ozs., canaga oil,  $8\frac{1}{2}$  ozs. bergamot oil, 14 ozs. lavender oil,  $2\frac{1}{2}$  drachms artificial musk.

*Jockey Club soap*.—Tallow stock soap 100 lbs. *Color*:  $3\frac{1}{2}$  ozs. cinnabar. *Perfume*: 7 ozs. artificial rose oil, 7 ozs. geranium oil, 9 qzs. eugenol, 2 ozs. hyacinthine, 0.7 ozs. artificial

cassia oil, 2 ozs. artificial jasmine oil,  $3\frac{1}{2}$  drachms artificial musk.

*White Rose soap.*—White stock soap 100 lbs. *Perfume:* 2 $\frac{1}{2}$  ozs. geranium oil,  $\frac{1}{2}$  oz. artificial rose oil, 1.4 ozs. artificial jasmine oil, 1.4 ozs. artificial oil of neroli,  $\frac{1}{2}$  oz. patchouli oil, 2 $\frac{1}{2}$  drachms civet.

*Rose soap.*—White stock soap 100 lbs. *Color:* 3 $\frac{1}{2}$  ozs. light cinnabar. *Perfume:*  $\frac{1}{2}$  oz. hyacinthine, 30 ozs. geranium oil, 0.7 oz. artificial oil of neroli,  $\frac{1}{2}$  oz. eugenol, 2 $\frac{1}{2}$  drachms civet, 1.4 ozs. lavender oil.

*Ess-bouquet soap.*—Palm oil stock soap 100 lbs. *Color:* 2 ozs. cinnabar, 14 ozs. caramel. *Perfume:* 12 ozs. bergamot oil, 13 ozs. artificial rose oil, 1.4 ozs. artificial jasmine oil, 10 $\frac{1}{2}$  ozs. lavender oil, 14 ozs. tincture of benzoin.\*

*Another formula* is as follows: Palm oil stock soap 100 lbs. *Color:* 1.4 ozs. cinnabar, 12 ozs. soap brown. *Perfume:* 6 $\frac{1}{2}$  ozs. bergamot oil, 14 $\frac{1}{2}$  ozs. geranium oil, 0.7 oz. artificial jasmine oil, 1 oz. hyacinthine, 7 ozs. eugenol, 8 $\frac{1}{2}$  ozs. tincture of benzoin, 7 ozs. tincture of tolu balsam†, 2 $\frac{1}{2}$  drachms civet ground up in a dish with 3 $\frac{1}{2}$  ozs. orris powder.

*Borax soap (milled)* White stock soap 80 lbs., 17 ozs. borax dissolved in boiling water. (*Cold stirred*) Cocoanut oil 48 lbs., lard 12 lbs., caustic soda lye of 38° B. 30 lbs., 21 ozs. borax dissolved in boiling water. *Perfume:* Bergamot oil 5 $\frac{1}{2}$  ozs., linaloe oil 4 $\frac{1}{2}$  ozs., lavender oil 1.4 ozs.

*Egg soap.*—White stock soap 50 lbs., *Color:*  $\frac{1}{2}$  oz. yellow aniline color. *Perfume:* 5 $\frac{1}{2}$  ozs. cassia oil, 4 $\frac{1}{2}$  ozs. citronella oil, 1 $\frac{1}{2}$  ozs. thyme oil, 0.8 oz. peppermint oil.

*Hygienic soap.*—White stock soap 200 lbs., lanolin 6 lbs., potato flour 1 lb. *Color:*  $\frac{1}{2}$  oz. light wax yellow. *Perfume:* 7 ozs. linaloe oil, 3 $\frac{1}{2}$  ozs. lavender oil, 2 $\frac{1}{2}$  ozs. canango oil, 2 $\frac{1}{2}$  ozs. spike lavender oil, 2 ozs. bergamot oil, 3 $\frac{1}{2}$  ozs. orange extract, 2 ozs. orris root tincture.

\* Tincture of benzoin is made from benzoin 1 lb. and alcohol 2 lbs.

† Tincture of tolu balsam is made from tolu balsam 1 lb., alcohol 3 lbs.

*Lanolin soap*.—White stock-soap 80 lbs., lanolin 2 lbs.

*Perfume*: 7 ozs. linaloe oil, 2 ozs. lavender oil, 1½ ozs. citronella oil.

*Almond blossom soap*.—Stock soap 100 lbs. *Perfume*: ½ oz. genuine bitter almond oil, 1 oz. cassia oil, 1½ ozs. terpineol, 5½ ozs. palma rosa oil.

*Milk soap*.—On account of their mild action upon the skin milk soaps may be recommended as toilet soaps although they do not allow of being stored for a long time as they easily turn rancid. A good quality of milk soap for milling is prepared as follows: Skim milk is evaporated in a vacuum apparatus to  $\frac{1}{2}$  its volume, at a low temperature, to avoid scorching and consequent darkening. The stock soap is then prepared as follows: Tallow 86 lbs. and Ceylon cocoanut oil 14 lbs. are dissolved in a kettle, and the melted fat is poured through a sieve which has been covered with a cloth to remove all impurities. The fat is then returned to the kettle and heated to 178° F., it being best to use a jacketed kettle and indirect steam. When the fat is sufficiently heated add slowly, crutching constantly, 71 lbs. soda lye and 2 lbs potash lye and then crutch the mass until it is thick. Now cover the kettle and allow the soap to combine so that it acquires a glassy appearance throughout. Then allow the soap to cool to 144° F. and fill it in the customary manner with evaporated milk; of the latter 50 per cent. may be added, but soaps with an addition of 25 per cent. are best for milling. When the milk has been thoroughly crutched in, the soap, in order to cool it as rapidly as possible, is poured into small shallow sheet iron frames holding from 50 to 60 lbs. each. The next day the soap may be cut, converted into shavings and dried when it is ready for milling.

*Orange blossom soap*.—Stock soap 100 lbs. *Perfume*: 1.4 ozs. neroli oil, 10½ ozs. lavender oil, 1½ ozs. bergamot oil, 1½ ozs. geranium oil, 1 oz. lemon oil, 3½ ozs. tincture of musk.

*Milled family soaps*.—For these soaps an inferior grade of stock soap may be used.

I. *Honey soap*.—Stock soap 60 lbs. *Color*:  $\frac{1}{2}$  oz. orange yellow, dissolved. *Perfume*: 3 ozs. citronella oil,  $2\frac{1}{4}$  ozs. lavender oil

II. *Windsor soap*.—Stock soap 60 lbs. *Color*:  $3\frac{1}{2}$  ozs. soap brown. *Perfume*:  $3\frac{1}{2}$  ozs. tincture of musk,  $2\frac{1}{4}$  ozs. oil of cloves,  $3\frac{1}{2}$  ozs. cassia oil,  $1\frac{1}{2}$  ozs. lavender oil.

III. *Patchouli soap*.—Stock soap 60 lbs. *Perfume*:  $\frac{3}{4}$  oz. patchouli oil,  $1\frac{1}{2}$  ozs. cassia oil, 1 oz. bergamot oil.

IV. *Violet soap*.—Stock soap 50 lbs., palm oil soap 10 lbs. *Color*:  $5\frac{1}{2}$  ozs. umber,  $\frac{1}{2}$  oz. brilliant brown. *Perfume*:  $2\frac{1}{2}$  ozs. bergamot oil,  $\frac{1}{2}$  oz. lavender oil.

V. *Musk soap*.—Stock soap 60 lbs. *Color*:  $5\frac{1}{2}$  ozs. umber,  $\frac{1}{2}$  oz. brilliant brown. *Perfume*:  $1\frac{1}{2}$  ozs. oil of cloves,  $\frac{1}{2}$  oz. wintergreen oil,  $\frac{1}{2}$  oz. patchouli oil,  $2\frac{1}{4}$  ozs. tincture of civet,  $3\frac{1}{2}$  ozs. vaselin,  $1\frac{1}{2}$  ozs. benzoin.

FLOATING SOAP.—The following directions may serve for the production of a white floating soap from fresh raw material, the color of the product of course depending largely on the color of the oil used: Cocoanut oil 88 lbs, caustic soda lye of  $38^{\circ}$  B. 43 lbs., caustic potash lye of  $29^{\circ}$  B. 2 lbs. Melt the cocoanut oil in the customary manner, strain it, if necessary, into a capacious jacketed kettle and heat to about  $122^{\circ}$  F. Then add the lye, stir thoroughly for about ten minutes, and cover the kettle. When saponification is complete, the soap is again crutched, and should then present a net-like, thick appearance. For further working but little fire or steam is required. Now gradually add in an uninterrupted stream 20 pounds of potassium chloride solution of  $20^{\circ}$  B. and 80 pounds of hot water. To promote solution the soap should be thoroughly stirred and slightly heated, but it is not necessary to bring it to the boiling point. When all is thoroughly dissolved, fill a glass cylinder with the soap solution, allow it to cool to  $77^{\circ}$  F., and test it with a hydrometer, which should indicate a density of about  $45^{\circ}$  B.; at this degree a medium heavy floating soap is obtained. Now allow the soap solution in the kettle to cool to  $77^{\circ}$  F. Enough of the soap is then brought into the stirring kettle to fill it one-third full so as to

leave plenty room for the expansion of the soap. The stirring kettle is provided with an arrangement for emptying it and with a stirring apparatus, the latter consisting of a steel shaft and steel blades, fixed so that it can be conveniently removed. The liquid soap in this kettle is then vigorously agitated or beaten until it is converted into a stiff froth, which is immediately brought into frames. The above-mentioned temperature must be strictly maintained, for, if the soap solution should have a higher temperature, for instance, 100° F., more time would be required to convert it into a permanent froth, and besides, the latter would, by long-continued stirring, become too loose so that the resulting soap would turn out too light and spongy. If, on the other hand, the soap solution is allowed to cool too much, the resulting soap would be too heavy because the formation of froth is not effected in a suitable manner. To obtain a somewhat more compact product, the soap in the frame may be covered with a suitable lid and the latter loaded with stones or weights. This, to be sure, reduces the volume of the soap, but it then contains fewer air-bubbles, becomes denser and can be more readily cut into bars and cakes. The soap should not be dried in a very warm room or in a drying oven, as otherwise it becomes cracked and gives a great deal of waste. However, the entire block, after being removed from the frame, should for several weeks be placed in an airy room which is not exposed to the rays of the sun. The blocks are then cut into slabs, which again are allowed to dry for several days, and then finally cut into bars and cakes.

*Floating soap from waste cuttings.*—The preparation of this soap is quite simple. Bring, for instance, 300 pounds of cuttings of a cocoanut oil soap into a jacketed kettle and, for their solution, add about 46 pounds of potassium chloride solution of 20° B., and about 200 to 240 pounds of water, the quantity of the latter depending on the degree of dryness of the cuttings. To promote solution the mass is at first quite strongly heated and frequently stirred. Large pieces of cut-

tings should, as a rule, be first reduced by a cutting machine. Very old and dry cuttings dissolve with difficulty; solution can, however, be accelerated by scattering 6 to 8 pounds of common salt over the above-mentioned quantity of cuttings in the kettle. Only cuttings from pure cocoanut oil soap should be used, those from filled soaps not being available, because such filling agents as soda and silicate of soda have a detrimental effect upon the product, the soap turning out brittle and harsh so that the surface of the cut bars and cakes do not present a smooth appearance. A similar effect is also caused by the use of common salt in too large quantities. When the soap in the kettle is thoroughly dissolved it is run through a fine sieve into another kettle, in order to remove particles of paper and wood as well as other impurities. The further operations are the same as previously described.

Many soapboilers maintain that cocoanut oil by itself is not very suitable for floating soap on account of its yielding a lather that is not sufficiently permanent. A paste soap of 250 per cent. yield has been recommended by L. Borchert. It should not contain too much water, neither should it be too dry, and is made according to the following quantities: Cocoanut oil 50 lbs., tallow 25 lbs., caustic soda lye of 36° B., 40 lbs. potassium carbonate solution of 20° B. 40 lbs., potassium chloride solution of 20° B. 20 lbs., brine of 20° B. 25 lbs.

Heat the cocoanut oil and tallow to 86° F., then stir in the 40 lbs. of caustic soda lye, allow the soap to stand 1 to 1½ hours to heat, and finally crutch in the potassium carbonate, potassium chloride and brine solutions, boiling hot, thus producing a paste soap. The soap is beaten in the stirring kettle as previously described. In the beginning only a sufficient quantity of it is brought into the kettle for the stirring apparatus to dip into it. When this is beaten into a froth the remainder is gradually added and beaten, the soap being kept meanwhile at about 194° F. At the end of the operation the soap will have increased more than twice its original bulk, and this must be taken into consideration in selecting the kettle for the process.

*Coloring floating soap.*—These soaps are, as a rule, preferred white, but, if required, red shades may be produced with cinnabar, brilliant rose, oriental rose and cardinal red, and yellow with water-soluble tampico yellow, tampico orange, etc.

*Perfuming floating soaps.*—The most useful perfumes are for white soaps: Lavender oil, caraway oil, white thyme oil and fennel oil; for rose-color or red soaps: Lavender oil, oil of cloves and palma rosa oil; for yellow soaps: Lavender oil and cassia oil.

Floating soaps are cut with a fine steel wire, and for smoothing the bars a sharp, finely-set plane is necessary.

*Shaving soaps.*—These soaps are prepared in various ways. The properties demanded from a good quality of shaving soap are, that it yields a good and heavy lather, which should remain standing for a considerable time, and at the same time be mild and delicate.

To combine these properties, fats yielding a good lather have to be chosen and saponified with alkalies exerting a mild effect upon the beard and skin. A neutral grained soap is the best to use, and if boiled with both soda and potash lyes, instead of soda lye alone, it is rendered still milder. While two-thirds of soda lye to one-third of potash lye are usually employed, it is better to take one-half of each. Soaps from cocoanut oil and palmnut oil yield an excellent lather, but the latter is not permanent enough. Tallow soaps, on the other hand, do not lather as freely, but the lather is more permanent. Hence a better lathering soap is obtained by saponifying some cocoanut oil with the tallow in the proportion of 10 to 12 lbs. of the former to 100 lbs. of the latter. The following composition may serve as an example.

Saponify 100 pounds of tallow with lye of 15° B. and boil clear. After removing the salty lye add 10 pounds of cocoanut oil and saponify it with potash lye of 25° B. When the whole is thoroughly combined, fit the soap slightly, and after allowing it to boil through, bring it into the frame. Perfume with  $3\frac{1}{2}$  ounces of oil of cassia and  $4\frac{1}{2}$  ounces of oil of thyme.

A shaving soap can be boiled both by the direct and indirect methods from a stock composed of: Tallow 70 parts, lard 20 parts, and cocoanut oil 10 parts with a lye made half of soda and half of potash. For the indirect process 100 parts of fat require about 300 parts of caustic lye of 10° B. Bring the tallow and lard into the kettle and begin saponification with caustic soda lye of 10° B. When the soda lye has completely combined run in the potash lye whilst the mass is briskly boiling. The paste is boiled up until samples taken out are solid and become moist. The paste soap is then saturated with caustic soda lye of 40° B. until separation of the grain takes place. Complete separation does not take place at once, it being attained by further steaming. When the lye runs clear from a sample taken out on the paddle, the kettle is tightly covered and the spent lye run off after it has separated. The stock soap is now very sharp, as a rule, and contains sufficient lye mixed up with it to completely saponify the cocoanut oil, which is now added. On heating the soap containing the cocoanut oil and adding brine of 4° B. or potassium chloride solution, it can be made like a separated grained soap. It is then always free from froth, but should it froth during separation, it will by further steaming boil clear. It must also be tested to see whether it is too sharp or not sharp enough.

When boiling a grained shaving soap from the same fats by the direct process, 100 pounds of fat will take about 100 pounds of caustic lye of 25° B. Heat the larger part of the lye, or half of the soda and half the potash lye in a kettle and run in the cocoanut oil and lard. A soap is formed which is in a condition to combine with the tallow subsequently added, so that when all the fats and lye boil in the kettle a soap resembling Eschweg soap is obtained. If the soap is inclined to become thick, strong potassium chloride solution is added. When properly fitted the soap should not acquire a glassy appearance, and a cooled sample should not be soft; if necessary lye is added until a sample when kept warm for some time is

solid and white. Potassium chloride solution is then added until the soap separates from the thick turbid lye.

*Cold-stirred shaving soap*—Bring into the kettle 80 pounds of white tallow and 40 pounds of cocoanut oil. Heat to about 99.5° F., and the fats being melted, add in the usual manner 64 pounds of caustic soda lye of 30° B. and 16 pounds of potash lye of 30° B., until the mass forms a well-combined, homogeneous paste. The entire operation requires at the utmost fifteen to twenty minutes. It is finished when the surface of the soap becomes covered with a film which constantly re-forms, notwithstanding stirring. Perfume the soap with oils of lavender and thyme, each 3½ ounces, oil of cumin 7 ounces, oil of bergamot 10½ ounces. The perfume is added to the soap, with constant stirring, before bringing it into the frame.

A few additional formulas for cold-stirred shaving soaps are here given:

I. Tallow 30 lbs., olive oil 14 lbs., cocoanut oil 6 lbs., caustic soda lye of 35° B. 20 lbs., potash lye of 10° B. 18 lbs. Heat the fats to 120° F. *Perfume*: Bergamot oil 2½ ozs., oil of caraway 1½ ozs., lavender oil 1½ ozs., thyme oil ½ oz., tincture of musk 2¾ drachms.

II. Tallow 34 lbs., oil of almonds 10 lbs., cocoanut oil 6 lbs., caustic soda lye of 25° B. 20 lbs., potash lye of 20° B. 19 lbs. Keep the fat at 116° to 118° F. *Perfume*: Bergamot oil 1½ ozs., oil of bitter almonds 2¾ drachms, geranium oil 3½ ozs.

III. Tallow 33 lbs., sesame oil 10 lbs., cocoanut oil 6 lbs., caustic soda lye of 35° B. 20 lbs., potash lye of 20° B. 18 lbs. Heat the fat to from 86° to 90° F. *Perfume*: Safrol 2 ozs., peppermint oil ½ oz., patchouli oil 2¾ drachms.

For the preparation of a shaving soap from tallow and almond oil by the cold process, Schimmel & Co., Leipzig, Germany, give the following formula: Tallow 85 parts and fatty almond oil 15 parts are stirred up at 113° to 115° F. with 25 parts of caustic soda lye of 38° B. and 25 parts of potash lye of 38° B. Saponification proceeds smoothly, and the resulting soap gives an excellent lather and has a fine white appearance. The bars do not warp, but remain rectangular.

*Shaving soap powders* are made as follows: First prepare a good shaving soap in the warm way from, for instance, 500 lbs. of tallow and 100 lbs. of cocoanut oil. Boil the fats with equal parts of caustic soda lye of 25° B. and caustic potash lye of 25° B. to a clear paste and separate the soap with brine. The settled grain is brought into frames and when cold, is cut into bars which are converted into shavings. The latter are thoroughly dried and ground to a fine powder in a mill. This powder is mixed with starch in the proportion of 15 to 20 lbs. of the latter to 100 lbs. of the former. By the addition of starch to the soap powder, a shaving powder is obtained which gives a fine permanent lather. Before mixing the starch with the soap powder it is perfumed, as a rule, with a mixture of lavender oil, thyme oil, caraway oil and fennel oil, an agreeable perfume being composed as follows: Starch 30 lbs., lavender oil  $2\frac{1}{2}$  ozs., lemon oil and thyme oil each  $1\frac{1}{2}$  ozs. The starch and perfume are intimately mixed and the whole is passed through a sieve which, however, should not be too fine. If the perfume is added to the powdered soap it forms small balls which in spite of all trouble cannot be entirely got rid of and finally remain behind upon the sieve.

A very fine shaving powder with a very durable and pleasant odor is obtained by a mixture of powdered soap 50 lbs., powdered orris root 8 lbs., and almond paste 4 lbs. The whole is intimately mixed and then passed through a sieve.

*Shaving pastes or creams.* The alkaline base of these preparations is potash and the fatty substances generally used are lard, olein, olive oil, sesame oil, and cocoanut oil. The following formulas may serve as examples:

I. Melt 20 pounds of lard on a water-bath at 212° F., add with constant stirring, 5 pounds of potash lye of 1.33 specific gravity, and keep the mixture at this temperature, gradually adding, with constant stirring, 5 pounds more of potash lye. Saponification is finished in a few hours. The cooled soap is made into a perfectly homogeneous paste by rubbing in a mortar, and for *crème d'amandes* perfumed with oil of bitter

almonds, and for *crème de rose* with oil of rose, and in the latter case colored rose-red.

II. Olive oil 60 lbs., olein 40 lbs., potash lye of 22° B. 100 lbs., caustic soda lye of 30° B. 5 lbs. Run half the lye into the kettle, allow to boil up, then gradually add the olein and when saponified, gradually introduce the remainder of the lye and the olive oil. When the soap is well boiled and fitted, a few per cents. of cocoanut oil or palm oil may be added. When the soap boils in light "flowers," free from froth, and a sample on cooling is salve-like and of a grayish color, the operation is finished.

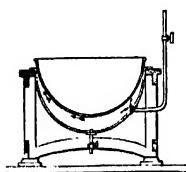
SOFT TOILET SOAPS OR SOAP CREAMS, are potash soaps. They serve chiefly for toilet purposes and must, therefore, be very delicate and mild. Prepare first a clear caustic potash lye of 20° to 21° B. Now bring 50 pounds of white lard and 10 pounds of cocoanut oil into a clean, roomy kettle; apply gentle heat, and, when the fat is melted, add 50 pounds of potash lye of 20° to 21° B.; keep the mixture at a temperature of from 167° to 189° F., and stir constantly. By the action of the heat and stirring, the aqueous portion of the lye is evaporated, and the mixture acquires a thicker consistency. It may happen that a portion of the fatty substances separates from the mass, which is chiefly the case when the temperature of the mixture is raised to a degree approaching the boiling point, because at this temperature concentrated lyes, with but few exceptions, have, as a rule, little affinity for the fatty substances. It may, however, also be due to the inadequacy of alkali in the mixture. In the first case, the combination is restored by moderating the heat, and, in the other, saponification is completed by pouring in a quantity of stronger lye.

For the first operation, which is called preparatory boiling, about four hours are required. For the complete saponification of the fat, about 30 pounds of potash lye of 35° B. are then gradually added, care being had to keep the mixture homogeneous by constant stirring, and keeping the temperature below the boiling point and as stationary as possible be-

tween 167° and 189° F. The end of saponification is recognized by the paste acquiring a very thick consistency. Stirring now becoming more difficult, the fire is removed, as, notwithstanding the stirring, the soap might settle on the bottom and scorch, which would not only change the quality of the soap, but impair its white color. Many manufacturers prepare this soap in an iron kettle, with double bottom heated by steam; some use silver kettles, which are preferable, because the soap will retain in them all its whiteness.

Fig. 53 represents a kettle with a double bottom heated by steam. This kettle is of tinned copper, and may be used also to purify tallow and greases.

FIG. 53.



The entire operation of preparing this soft soap lasts from seven to eight hours. When the soap is cool, it has a soft and pasty consistency, and is poured into large stone or porcelain jars in which it is kept for use.

Soft soap, as obtained by the saponification of fatty substances by potash, has not that bright and nacreous appearance required for the toilet. To attain this state, it is ground in a marble mortar, and aromatized with oil of bitter almonds, about 1 ounce being sufficient for the above quantity.

Although oil of bitter almonds is principally used as a perfume for these *soap creams*, other fragrant substances may be employed.

*Liquid toilet soaps.* These products are simply solutions of soap which must, however, be carefully prepared to possess the required qualities. The liquid soap should, in a somewhat thicker layer, appear as a translucent, opalescent mass of such a consistency that it flows from a bottle in a viscid stream like thin honey.

Perfectly pure white hard soap free from odor has to be used for the preparation of liquid soap. The soap is converted into fine shavings and the latter are gradually brought into

boiling water until a cooled sample shows the above-mentioned properties. The liquid is then allowed to boil gently and frequently stirred to bring every particle of soap into solution and at the same time obtain a homogeneous fluid. Stirring is continued until the fluid shows the peculiar opalescent appearance characteristic of concentrated soap solutions.

The finished soap is allowed to cool to the ordinary temperature when the perfume and coloring matter are added. Aniline colors are used for coloring and should be dissolved in the smallest possible quantity of alcohol free from fusel oil. The soaps are colored according to the perfume used, for instance, pale red for rose soap, pale violet for violet soap, etc. However, the coloring matter should be used sparingly, so that the soap shows the color only in thicker layers, but appears almost colorless in thinner layers. On the other hand, quite a large quantity of perfume may be used so that the hands are perceptibly scented after washing with the soap.

After adding the perfume and coloring matter, the fluid is again stirred at the ordinary temperature until it is uniformly colored and scented.

The finished liquid soap is at once brought into the bottles in which it is to be sold.

*Liquid glycerin soaps.* I. Heat in an enameled vessel on a water-bath, olein 250 parts by weight, stir in 750 parts by weight of glycerin, and heat the mixture to 140° F. Then run in 100 parts by weight of caustic potash lye of 38° B. previously diluted with 25 parts by weight of distilled water, when saponification will immediately take place. Allow the mass, which is quite thick, to cool, and then stir in a solution of 15 parts of potassium carbonate in 25 parts by weight of hot water. Now cover the vessel and allow the soap to stand two to three days. Then pour off the clear soap from the sediment, add the perfume dissolved in alcohol, and after well shaking the whole allow it to stand quietly for some days. Finally filter the soap through paper and fill it in bottles for use. Filtration being rather slow the soap should be well

covered to prevent the perfume from evaporating. Any desired perfume may be used, but the essential oils must be dissolved in 96 per cent. alcohol.

II. Dissolve 11 parts of caustic potash in sticks in 30 parts of alcohol, add 60 parts of peanut oil, and allow the whole to stand at about 86° F. until saponification is complete. Dissolve the solid soap formed in equal parts by weight of glycerin. Peanut oil is more suitable than olive oil, as it saponifies more quickly and yields a finer soap. With the use of pure materials filtering is not necessary.

III. Melt 35 lbs. Cochin cocoanut nut oil and 35 lbs. cotton-seed oil, strain the melted fat and, at 178° F., stir in in a thin stream 80 lbs. caustic potash lye of 20° B., crutch thoroughly, and add 130 lbs. glycerin of 24° B. and 50 lbs. water. Cover the kettle for half an hour and then stir in 120 lbs. 95 per cent. alcohol. *Perfume*:  $\frac{1}{2}$  oz. palma rosa oil,  $\frac{1}{2}$  oz. each of oil of cloves,  $\frac{1}{2}$  oz. each of lavender oil and bergamot oil and 2 $\frac{1}{2}$  drachms tincture of musk.

IV. Mix white potash soap 1 oz., glycerin 3 ozs., sugar syrup 1 oz., 90 per cent. alcohol  $\frac{1}{2}$  oz., cinnamon oil, geranium oil, sassafras oil and oil of cloves 2 drops each, citronella oil 5 drops, wintergreen oil 2 drops, tincture of musk 1 drop. Let the mixture stand for a few days, and filter.

V. Dissolve Venice soap in shavings 2 ozs., and pure potash 2 $\frac{1}{2}$  drachms in alcohol 17 ozs., and glycerin 10 $\frac{1}{2}$  ozs., and add 7 ozs., more of alcohol to the solution. Filter, and then add 1 drachm each of lemon and bergamot oils.

*Medicated soaps*.—Every soapmaker engaged in the preparation of medicated soaps is aware of the painstaking care and accuracy required to make soaps according to a physician's prescription and, although many manufacturers turn out an excellent product there are many so-called medicated soaps brought into commerce which do not deserve the name, since they frequently contain not even a trace of efficacious substances. Again, we find other medicated soaps which are inoperative on account of ignorance as regards the decomposi-

tion of the chemical agents added to the soap. In many iodine soaps, for instance, a small addition of potassium iodide is found, but no sodium hyposulphite, which is absolutely necessary to render the iodine efficacious. And, thus there are many other soaps, which from a want of sufficient chemical knowledge are not properly composed, and instead of having a soothing and healing effect in skin diseases promote the diseased state.

Medicated soaps are prepared either in the cold or warm way, similar to milled soaps.

Dr. P. G. Unna has pointed out the importance of soap as a remedy in dermatology. He prepares his medicated soaps in accordance with the following principles: He employs only one fat, namely, the best quality of beef tallow, and rejects coconut oil entirely, on account of its roughening effect on the skin when continuously used. The alkali employed consists of a mixture of 2—in the middle of summer 3—parts of freshly-prepared soda lye and 1 part of potash lye, the total quantity being just sufficient to leave the saponified mass perfectly neutral in reaction. These soda-potash soaps are solid, more efficacious than soaps from soda alone, and do not peel like the latter sometimes do. As neutral soaps, when permanently incorporated with the skin, abstract fat therefrom, and produce a disagreeable brittleness attended by congestion and roughness, Dr. Unna superfats his soaps by taking, beyond the amount of fat necessary for saponification, an excess—averaging 3 to 4 per cent.—of fat, consisting of 8 parts tallow and 1 part olive oil. These superfatted soaps have the additional advantage of better preserving sundry medicaments, such as salicylic acid, unstable salts and sublimate better than ordinary neutral soaps. Of course, all the other conditions applicable to the best toilet soaps must also be fulfilled. Dr. Unna recommends the following formula for a superfatted stock soap:

Best beef tallow 16 parts, olive oil 2 parts, caustic soda lye of  $38^{\circ}$  B. 6 parts, caustic potash lye of  $38^{\circ}$  B. 3 parts. The most interesting of Dr. Unna's medicated soaps are the super-

fat salicylic soap and super-fat zinc salicylic soap. These soaps prepared from the best salicylic acid are used :—

1. As disinfecting soaps in all fungoid affections of the skin.
2. As an auxiliary remedy in the form of simple ablutions, with water as hot as possible in severe, obstinate and strongly itching cases of eczema.
3. In acne partially to promote the removal of the diseased horny layer and to lay bare the follicle closed by it, and partially to remove the black horny points of the comedones.

The composition of the

*Superfat salicylic-soap* is as follows : Super-fat stock-soap 95 parts, salicylic acid 5 parts. The soap is yellowish white and quite soft, and inclined with repeated soaking and drying to become brittle, and should, therefore, be kept dry.

*Superfat zinc salicylic-soap* is composed of 88 parts of stock-soap, 2 parts of zinc oxide, and 10 parts of salicylic acid. This soap is white, very hard, yields little lather with cold water, but an abundant one with hot water, with which it is directed to be used.

Other super-fat soaps prepared according to Dr Unna's directions are :—

*Superfat tar-soap* contains 5 per cent. of tar.

*Superfat sulphur-soap* contains 10 per cent. of precipitated sulphur.

*Superfat tar-sulphur soap* contains 5 per cent. each of tar and precipitated sulphur.

*Superfat camphor-sulphur soap* with 5 per cent. of camphor and 10 per cent. of precipitated sulphur.

*Superfat camphor-soap* with 5 per cent. of camphor.

*Superfat borax-soap* with 5 per cent. of sodium borate.

*Superfat iodine-soap* with 5 per cent. of potassium iodide.

*Superfat naphthol-soap* with 5 per cent. of naphthol.

*Superfat naphthol-sulphur soap* with 5 per cent. each of naphthol- $\beta$  and precipitated sulphur.

*Other Formulas for Medicated Soaps.—Arsenic soap* is chiefly used for preserving skins of animals for stuffing purposes by

application to the flesh side. Mix intimately 10 ozs. salicylic acid, 5 ozs. powdered borax, 5 ozs. powdered naphtholene, 20 ozs. ground arsenious acid, 50 ozs. soap and 100 ozs. water. Apply the salve-like soap to the flesh of the skin.

Another formula is as follows:—Heat until all is dissolved, 11 ozs. arsenic, 4 ozs. potassium carbonate and 11 ozs. water. Then add 11 ozs. Marseilles soap in shavings, stir the whole and when dissolved, introduce 1½ ozs. quick lime and ½ oz. powdered camphor.

*Pumice soaps.*—I. *For disinfecting the hands, etc.*—According to L. Pförringer a neutral soap made from a vegetable fat is best suited for this purpose. Cut into fine shavings 3 ozs. of soap—the use of a larger quantity is not advisable as the resulting product would turn out too hard. Dissolve the shavings in 300 ccm. of 96° to 97° per cent. alcohol on a hot water bath with an inverted condenser. When solution is complete make up the quantity to 1000 ccm. by the further addition of 700 ccm. of hot 96 per cent. alcohol. Then add gradually 10½ ozs. of finely powdered pumice previously dried and sterilized and allow the mixture to cool slowly, shaking it vigorously all the while in order to distribute the pumice as uniformly as possible. If insufficiently shaken two layers form after cooling, a lower one of pumice and an upper one consisting of alcoholic soap emulsion. During this shaking the soap solidifies to a cream; it becomes subsequently more solid and can in this consistency be poured out. The soap should be kept in air-tight vessels otherwise the alcohol gradually evaporates.

While, strictly speaking, pumice soaps cannot be called medicated soaps, they are useful as a mechanical agent for removing warts and callosities. They are generally prepared by the cold process from cocoanut oil alone or in combination with tallow, cottonseed oil, bleached palm oil, etc. The oil is melted and the lye stirred in at a temperature of 89° to 95° F. The finely powdered pumice is then sifted in and the soap perfumed. The following compositions may serve as examples:

II. Cocoanut oil 80 lbs., cottonseed oil 20 lbs., caustic soda lye of 38° B. 48 lbs., caustic potash lye of 30° B. 2 lbs., pulverized pumice 50 lbs. *Perfume*: Cassia oil 5½ ozs., rosemary oil 3½ ozs., lavender oil 1½ ozs., safrol 1½ ozs., oil of cloves ½ oz.

III. Cocoanut oil 100 lbs., caustic soda lye of 40° B. 50 lbs., powdered pumice 100 lbs. *Perfume*: Lavender oil 8½ ozs., caraway seed oil 2½ ozs.

A bluish-gray shade, which is frequently desired, may be imparted by a little ultramarine.

*Benzoin soap (in the cold way).* Prepare first by the cold way a good non-caustic soda soap from a mixture of cocoanut oil and lard, strip the soap, sprinkle the shavings with the color, then pour the benzoin tincture over them, mix in the mill, press, dry and mould the finished soap.

For benzoin soap use: 100 pounds of soap prepared as above and tincture of benzoin 8 pounds. Color brown with sugar color.

Benzoin tincture is prepared by treating a good quality of pulverized benzoin with alcohol. Benzoin soap has an agreeable vanilla-like odor.

*Camphor soap.* I. *Milled.* White stock soap 80 lbs., camphor finely pulverized and sifted 6 lbs.

II. *Cold stirred.* Cocoanut oil 40 lbs. and caustic soda lye of 38° to 40° B. 20 lbs., stirred together at 106° F., camphor 21 ozs. The camphor is dissolved in the oil, though it may also be dissolved in alcohol, and the solution crutched into the finished soap.

If the odor of camphor is to be covered, the soaps may be perfumed with sassafras oil and rosemary oil each 5 ozs., thyme oil 2½ ozs.

Camphor soap is used for chilblains, and in the bath for rheumatic pains.

*Carbolic soap.* I. *Milled.* White stock soap 80 lbs., chemically pure carbolic acid 2 lbs. stirred up in potash solution 2 lbs.

II. *Cold stirred.* Cocoanut oil 40 lbs., caustic soda lye of 38° B. 20 lbs., chemically pure carbolic acid 26 ozs. dissolved in potash lye 26 ozs. and glycerin 8½ ozs.

Carbolic soap serves for the removal of sycosis (barber's itch), dandruff, etc., as well as for general toilet use, especially during the prevalence of infectious diseases. For disinfecting linen a carbolic neutral potash soap is recommended, 3 per cent. of carbolic acid incorporated in the soap having a weak, and 5 per cent. a strong action. It may be prepared by stirring 10 lbs. liquid carbolic acid into 200 lbs. of unfilled soft soap, and eventually fitting with a small quantity of lye.

*Carbol-glycerin soap.*—Tallow, cocoanut-oil, and lye of 38° B. each 30 pounds, alcohol 15 pounds, liquid carbolic acid and glycerin each 12 pounds, sugar color ½ pound. Prepare in the same manner as glycerin soap.

*Creolin soap.* I. *Milled.* White stock soap 80 lbs., creolin 1 lb.

II. *Cold stirred.* Cocoanut oil 60 lbs., caustic soda lye of 38° B., 31 lbs., creolin 3 lbs. The creolin is dissolved in the cocoanut oil. Creolin soap has an antiseptic action.

*Iodine soap.* I. *Milled.* White stock soap 40 lbs., potassium iodide finely powdered and sifted 4 lbs.

II. *Cold stirred.* Cocoanut oil 30 lbs., caustic soda of lye 38° B. 15½ lbs., potassium iodide 4½ lbs. dissolved in as little water as possible.

Iodine soap is used for certain ailments of the hands and feet and also for the preparation of baths as a remedy for eruptions of the skin.

*A soap used for iodine and bromine baths* is prepared as follows:

Liquefy 10 lbs. best quality olein soft soap on the water bath and add a solution of 14½ ozs. liver of sulphur, 17½ ozs. potassium iodide, 10½ ozs. potassium bromide and 4½ ozs. pure ferrous sulphate (copperas). The whole is thoroughly agitated and then evaporated so that on cooling a firm soap results.

*Ichthyol soap.* I. *Milled.*—White stock soap 40 lbs., ammonia ichthyol ¼ lb.

II. *Cold stirred.*—Cocoanut oil 30 lbs., caustic soda lye of 38° B. 15½ lbs., ammonia ichthyol ¼ lb., dissolved in as little water at possible. Ichthyol soap is used for chilblains and various skin diseases.

*Sulphur soap.* I. *Milled.*—Yellow stock soap 40 lbs., liver of sulphur 4 lbs., dissolved in as little water as possible, are mixed in the customary manner in the mixing machine.

II. *Cold stirred.*—Cocoanut oil 20 lbs., caustic soda lye of 38° B. 10½ lbs., liver of sulphur 3 lbs. dissolved in water, 3 lbs.

*Tannin soap.* I. *Milled.*—White stock soap 80 lbs., tannic acid 2 lbs.

II. *Cold stirred.*—Cocoanut oil 60 lbs., caustic soda lye of 38° B. 31 lbs., tannic acid 1½ lbs., dissolved by heating in water 3 quarts. *Perfume:* Lavender oil, geranium oil and bergamot oil each 1 oz., peppermint oil 1½ ozs.

With stirred soap the tannic acid solution should be added at the latest possible moment, since with the admission of air tannic acid in the presence of free alkali rapidly decomposes. Tannin soap is recommended for herpes (ring-worm, etc.).

*Tar soap.* I. *Milled.*—Brown stock soap 40 lbs., wood tar 5 lbs.

II. *Cold stirred.*—Cocoanut oil 20 lbs., caustic soda lye of 38° B. 10¾ lbs., wood tar 2 lbs.

The stirred soap is prepared in the customary manner by stirring together the lye and cocoanut oil, the tar having been previously dissolved in the oil.

*Tar-sulphur soap.* I. *Milled.*—Yellow stock soap 40 lbs., liver of sulphur 4 lbs., wood tar 4 lbs.

II. *Cold stirred.*—Cocoanut oil 20 lbs., caustic soda lye of 38° B. 10½ lbs., liver of sulphur 3 lbs., wood tar 3 lbs.

Tar soaps are recommended for eruptions of the skin, dandruff, etc.

*Turpentine soap.*—Melt on a water bath white stock soap 6 lbs., and unfilled soft soap 6 lbs. When thoroughly liquefied, add and mix intimately 12 lbs. turpentine and 21 ozs.

finely powdered potash. The resulting product is of salve-like consistency and is filled into shallow glass boxes. It is highly recommended as an embrocation for rheumatism, gout, swellings, chiblains, etc.

*Naphthol soap.* I. *Milled.*—Stock soap 18 lbs., beta naphthol 1 lb.

II. *Cold stirred.*—Cocoanut oil 20 lbs., tallow 5 lbs., caustic soda lye of 38° B. 14 lbs., beta naphthol 7½ lbs. *Perfume:* Peppermint oil 2½ drachms, oil of cloves ½ oz., cassia oil ½ oz.

Stir the lye into the fat at 95° F., and when combination has been effected mix in the naphthol, then the perfume, and finally frame the soap and cover it up well. Naphthol soap is used for skin diseases.

*Naphthalene soap.*—Cocoanut oil 10 lbs., castor oil 2½ lbs., tallow 2½ lbs., caustic soda lye of 37° B. 8 lbs., naphthalene 1 lb. Stir the naphthalene into the fat heated to 98° F. and when the temperature has sunk to 86° F. add the lye. *Perfume:* Cassia oil 0.8 oz., peppermint oil 2½ drachms. The soap is recommended for excessive perspiration.

*Vaseline soap.* I. *Milled.*—White stock soap 40 lbs., white vaseline 3½ lbs. *Perfume:* Bergamot oil 3½ ozs., cinnamon oil ¾ oz., rose geranium oil 3½ ozs., oil of cloves ½ oz., oil of bitter almonds ½ drachm, Peruvian balsam and tincture of vetiver 3½ ozs. each.

II. *Cold stirred.*—Cocoanut oil heated to 86° F. 20 lbs., white or yellow vaseline 6 lbs., caustic soda lye of 38° B. 12 lbs. *Perfume:* Sassafras oil 1½ ozs., lavender oil 1 oz., rosemary oil ½ oz., oil of bitter almonds and oil of cloves 2½ drachms each. Stir the lye into the fat at 77° F.

*Thymol soap.* I. *Milled.*—White stock soap 40 lbs., thymol 1 lb., dissolved in a small quantity of alcohol.

II. *Cold Stirred.*—Cocoanut oil 30 lbs., caustic soda lye of 38° B. 16 lbs., thymol 1 lb., dissolved in a small quantity of alcohol. Thymol soap is recommended for diseases of the skin, its antiseptic properties being thus brought into effect.

## CHAPTER XVIII.

### ESSENTIAL OILS AND OTHER MATERIALS USED FOR PERFUMING SOAPS.

AN extensive class of bodies, nearly all limpid liquids, but some viscid, and a few solid, are known under the collective term of essential or volatile oils. Most of them are of vegetable origin, the animal kingdom furnishing but a few substances and of these only three, namely ambergris, civet and musk are used for toilet soaps. By the extraordinary progress which has of late been made in the chemistry of essential oils and the establishment of the chemical composition of many of them, a considerable number of important perfumes are now at the disposal of the soapmaker and perfumer. They are in part separated from the oils and presented to the consumer in a pure form, and partly prepared artificially. The most important of these products will be referred to later on.

The essential oils as found in commerce vary very much in quality, and are frequently adulterated and generally by simply mixing an expensive oil with a cheap one. Alcohol is also used, more rarely chloroform and fatty oil. In addition to these old customary means, the hydrocarbon, known as terpene, which is separated out in the production of concentrated oils, has recently come into use for adulterating purposes.

Whilst, thanks to the development of the chemistry of the terpenes during the last 10 to 15 years, great progress has been made in detecting adulterations, the incomplete knowledge of the composition and the lack of methods of examination admit at present, in the case of most of these oils, of no dependable tests based on rational chemical foundations, and for the

soapmaker it is therefore the safest plan to purchase these expensive articles from a source known to be reliable.

The substances which are mostly in use for perfumery purposes will be described below, together with the most important methods of testing them.

*Essential oils.* As no feasible classification of these oils has yet been discovered, they have for the sake of convenience been alphabetically arranged.

*Angelica oil* is obtained from the fruit as well as the root of *Angelica (Archangelica officinalis Hoffm. = Angelica Archangelica)*. The oil usually used is obtained from the root, but that from the fruit (seed) is of finer quality. When fresh the root oil is a mobile, colorless liquid which turns yellow to brown on exposure to light and air. It has an aromatic odor and burning taste. Its specific gravity ranges from 0.855 to 0.905 at 59° F. When exposed to the air the oil absorbs oxygen and gradually resinifies.

The seed oil possesses an odor similar to that of root oil, but much finer. Its specific gravity is 0.8549 at 59° F. Older oil is somewhat more thickly fluid, of a brown color, and specifically heavier.

*Anise oil* is distilled from the seeds of the anise plant, *Pimpinella anisum L.* It is colorless to pale yellow, is liquid at a higher temperature, but when cooled solidifies to a snow-white crystalline mass. A good quality of oil should solidify at from 41° to 59° F., and completely liquefy again at 42.8° to 64.4° F. However, if the oil be several times heated to its boiling-point it loses the capacity of crystallizing. The specific gravity of fresh oil is 0.98 to 0.995; that of old oil 1.0285. Anise oil gives a clear solution with 5 parts of 90 per cent. alcohol and 3.5 times its volume of petroleum-ether, a larger amount of the latter solvent giving a turbid mixture. It shows a neutral reaction, but air renders it resinous, and it then loses the property of crystallization. The odor of the oil is anise-like and its taste agreeable, sweetish and afterwards pungent. It should be stored in a dark, cool place in well-stoppered bottles, filled up to the neck.

*Star anise oil* is obtained from the fruit of a species of *Illicium* indigenous to China. It is very similar to the ordinary anise oil, having an anise-like odor and an intensely sweet taste. At 59° F. its specific gravity is 0.98 to 0.99. It dissolves to a clear solution in three or more parts of 90 per cent. alcohol.

Anise oil is adulterated with turpentine oil, cedar wood oil, copaiba and gurjun balsam oil, spirit, spermaceti and fatty oils. Such adulteration can be detected by the specific gravity and solidifying point. The oil is also adulterated with the fennel-stearoptene, which is detected by the peculiar odor when heating the suspected sample.

*Basil oil* is obtained by distilling with water sweet basil, *Ocynum Basilicum*, L. It possesses an aromatic odor and on standing separates prismatic crystals of a stearoptene. Its color is yellowish and the specific gravity ranges between 0.918 and 0.928.

*Bergamot oil* is obtained from the rinds of the *Citrus bergamia*, or bergamot-orange. It is of a pale green or yellowish color, becoming, however, darker with age. The specific gravity varies between 0.856 and 0.888 at 59° F. and the boiling point between 365° and 397.5° F. It congeals at 11° F. It is one of the most changeable oils and soon acquires an odor of turpentine. It is frequently adulterated with alcohol, being very soluble in it. For the detection of alcohol the following method is recommended by Righini: Mix 15 parts of bergamot oil with an equal quantity of olive oil or of sweet almond oil. If alcohol is present in the bergamot oil, it separates immediately from the fatty oil as though it were water. If the mixture does not separate it is certain that the oil is not adulterated with alcohol. Oil of bergamot is also adulterated with turpentine oil, orange-peel oil; fatty oils, cedarwood oil, but chiefly with lemon oil, such sophistications being recognized by variations in the specific gravity. From other oils of the family *Aurantiaceae* it differs by its ready solubility in alcohol, 1 part oil in  $\frac{1}{2}$  part alcohol.

*Bitter almond oil.*—This oil does not exist ready formed in the almond, but is a product of the decomposition of the bitter substance—amygdalin—of which the bitter almond contains 5 per cent. This substance is a glucoside and is decomposed in the presence of water and of a peculiar albuminoid ferment present in the almond and known as *emulsin*, into glucose, bitter almond oil, and hydrocyanic acid. The presence of the latter renders the crude oil of bitter almonds extremely poisonous. It can be freed from a large portion of the acid by shaking with lime, and completely so by the following process: Shake up the oil with a solution of iron and milk of lime, and then distil it over burnt lime, whereby it loses its original yellowish or yellow color and is rendered colorless.

Bitter almond oil is obtained by submitting bitter almond cake, which is left after the expression of the fixed oil from bitter almonds, to distillation with water. The press-cakes from the kernels of the apricot are also used for the production of bitter almond oil, the oil thus obtained being in no way distinguishable from that from bitter almonds.

In the crude state, bitter almond oil is yellowish to yellow, but when rectified forms a colorless, highly refractive liquid which on exposure to the air becomes viscid, acquires a yellow color and, in consequence of the absorption of oxygen, separates crystals of benzoic acid. The oil has a strong odor of bitter almonds and an aromatic, agreeable, somewhat pungent taste. Its specific gravity at 59° F. is 1.043; it boils at 354° F. It should be stored in a dark place in well stoppered bottles, filled to the neck.

Oil of bitter almonds is much adulterated with cheaper oils, but chiefly with nitrobenzole, which has an odor very similar to that of genuine oil. Pure oil of almond mixed with sulphuric acid gives a clear crimson-red color without perceptible decomposition; with an alcoholic solution of potash crystals are eliminated. The oil will take up as much as one-third of its weight of iodine, and retain the same in solution. Potassium chromate does not effect the oil. To detect the presence of

nitrobenzole, dissolve 1 part by weight of the suspected oil in 10 parts by weight of alcohol; compound the solution with 1.5 parts by weight of solid caustic potash, and evaporate the mixture to one-third its original volume. Pure bitter almond oil turns brown, but remains fluid, while, if adulterated with nitrobenzole, a brown resinous substance is formed, which floats in the liquid.

*Artificial oil of bitter almonds* is now extensively prepared as a substitute for the genuine bitter almond oil, and much used for scenting soaps, especially the cheaper varieties. It is known in trade as *nitrobenzole, oil of mirbane, essence of mirbane*, etc. Although it is not a natural essential oil, being an artificial product, it is considered best to refer to it here in connection with bitter almond oil. Artificial oil of bitter almonds boils at 408° F., and its specific gravity at 32° F. is 1.2.

*Cananga oil* is an inferior quality of ylang-ylang oil, and will be referred to when speaking of the latter.

*Caraway oil* is obtained by distillation from the seeds of the common caraway plant, *Carum carvi*. The oil is nearly colorless, of a very aromatic odor and acrid taste; it becomes yellow-brownish by age and then shows an acid reaction. It has a specific gravity varying between 0.900 and 0.960 at 59° F., and boils at from 347° to 446° F. It shows but little reaction with iodine, and gives an almost clear liquid with alcohol and sulphuric acid. When pure it is soluble in an equal volume of 90 per cent. alcohol; otherwise it contains turpentine oil, or does not possess the full amount of carvol, the compound contained in the oil which imparts to it the caraway smell.

*Cassia oil* is obtained by distillation from the buds and barks of *Cinnamomum cassia*, a tree indigenous to China. It is thickly fluid, of a golden-yellow color, and has a sweet, afterwards burning, taste. Its specific gravity ranges from 1.055 to 1.065. Its fragrance is pleasant and cinnamon-like, but less refined than that of the genuine cinnamon oil, under

which name it is frequently brought into commerce. Cassia oil brought into trade from China is said to be frequently adulterated with resin and petroleum. Such an adulteration can be readily detected by the specific gravity.

*Cedar-wood oil* is obtained by distillation with steam from the finely rasped wood of the Virginia cedar (*Juniperus virginiana* L.) and the waste obtained in the manufacture of lead pencils. At an ordinary temperature cedar-wood oil is a white crystalline body of the consistency of butter. The actual essential oil separated by pressing is colorless, very limpid, and solidifies only at a very low temperature, but readily resinifies on exposure to the air. Its specific gravity is 0.9622 at 59° F.; it boils at 519.8° F. and solidifies at 6° F. On account of its agreeable odor it is used as a basis for other perfumes for toilet soaps.

*Cinnamon-oil (genuine)* is obtained from the bark of the young branches of *Cinnamomum zeylanicum*, a tree cultivated in extensive plantations in Ceylon. The oil is heavier than water, its specific gravity being 1.005 to 1.050 at 59° F. It is of a pale-yellow to red-brown color, the latter indicating old oil, and has an agreeable odor and biting, but pure sweet taste. It remains liquid at—13° F. It gives a clear solution with 3 parts of 70 per cent. alcohol. Cinnamon oil is frequently adulterated with inferior qualities of cinnamon oil, such as cinnamon leaf oil, especially with cassia oil, further with alcohol, chloroform, oil of cloves, etc. A test for the purity of cinnamon oil is as follows: 1. Cinnamon oil, when allowed to fall drop by drop into cold water, should sink. 2. When placed upon the tongue it should immediately produce a taste of intense sweetness, exceeding the sweetness of sugar.

*Citronella oil* is obtained by distillation from *Andropogon Nardus* L. a grass grown in Southern Ceylon and Malacca, as well as in India. The oil is limpid, greenish yellow to brown, and has an agreeable odor which, however, comes out in its entire purity only when the oil is much diluted. The specific gravity of the oil ranges from 0.893 to 0.897 at 60° F. It

boils at 415.4° to 426.2° F. Pure citronella oil dissolves clear in 10 parts of 80 per cent. alcohol, and at 59° F. its specific gravity should not be below 0.895. It is frequently adulterated with fat oils as well as with petroleum. Such adulterations can be readily detected by the above-mentioned alcohol test which can be most suitably applied by means of graduated measure. The specific gravity of 80 per cent alcohol is 0.8645. If the sample is adulterated with fixed oil or petroleum, the mixture becomes cloudy, and drops of the adulterant are either precipitated or rise to the surface after the mixture has been allowed to stand for 12 hours.

*Cloves, oil of.*—Every part of the clove-tree, *Caryophyllus aromaticus*, abounds with aromatic oil, but it is most fragrant and plentiful in the unexpanded flower-buds which are the cloves of commerce. They are soaked for some time in salt water and then submitted to distillation. The oil, when fresh, is almost colorless, but on exposure to the air acquires a brownish color and a thickly-fluid consistency. It has the aromatic taste and odor of cloves. Its specific gravity ranges from 1.045 and 1.070 at 50° F.; it boils at 482° F. It is soluble in 2 parts of 70 per cent. alcohol. It is frequently adulterated with inferior oils. Such sophistication is detected by the specific gravity being lowered.

*Eucalyptus oil* is obtained from the leaves of various species of *Eucalyptus*. The oil obtained by distillation from the leaves of *Eucalyptus globulus* indigenous to Tasmania, but also much grown in southwestern Europe, is limpid, almost colorless and possesses a camphor-like odor. Its specific gravity ranges from 0.91 to 0.93 at 59° F.

*Fennel-oil* is derived by distillation from the seeds of the sweet fennel, *Foeniculum dulce*. When pure the oil is colorless, has a hot, pungent taste, and the odor of the plant. Its specific gravity is 0.965 to 0.975 at 59° F.; it boils at from 365° to 374° F. and congeals at from 39° to 64.5 F. It is soluble in an equal volume of 90 per cent. alcohol and in 5 to 8 volumes of 80 per cent. alcohol. It is rarely adulterated, but

frequently deprived by fractionating or by freezing of part of its anethol of which it contains 50 to 60 per cent.

*Geranium oil*.—The various oils of geranium known as African, Spanish, French and Réunion, are obtained from the leaves of several species of *Pelargonium* by distillation with water. There is but little or no difference in the properties of the various kinds of oil found in the market. Geranium oil is colorless, or greenish or brownish and smells very much like attar of roses, it being, for this reason, very frequently used for adulterating rose oil. The specific gravity of French oil is 0.897 to 0.95; that of African oil 0.892 to 0.90; that of Réunion oil 0.889 to 0.895; and that of Spanish oil 0.897. The three first mentioned oils form a clear solution with 2 to 3 parts of 70 per cent. alcohol. With Spanish oil the solution in one or more parts of 70 per cent. alcohol is generally cloudy through the presence of minute crystals of paraffin which, on continued standing, collect on the surface. The separation of drops of oil at the bottom of the vessel in the Spanish oil would denote adulteration with fatty oils.

Geranium oil is frequently adulterated by grass oils, further by copaiba oil, fatty oils and cocoanut oil. To detect such additions shake 5 drops of the oil with 5 cubic centimeters of 70 per cent. alcohol, whereby pure geranium oil should be dissolved. Furthermore grass oil is colored brown by iodine, while geranium oil is not.

*Juniper oil* is obtained by distillation from the juniper berry, the fruit of *Juniperus communis* L. Pure juniper oil is colorless, thinly-fluid, with a sweetish turpentine-like odor and taste. It has a specific gravity of 0.865 to 0.885, boils at 302° to 359° F., and the larger portion of it congeals on exposure to a low temperature. It is soluble in all proportions in glacial acetic acid. It is frequently adulterated with turpentine oil, which is, however, readily detected by the lessened density and solubility in alcohol.

*Lavender oil*.—The genuine lavender oil is distilled from the flowers of *Lavandula officinalis* Chaia; that produced in Eng-

land possesses the finest fragrance. When freshly prepared it is a colorless liquid, which becomes yellow on standing. It has a hot, camphorous, slightly bitter taste, and the odor of lavender. It has an acid reaction, a specific gravity of 0.885 to 0.895 at 59° F., and begins to boil at 365° F., the temperature quickly rising to 374° F., and the greater portion distilling over between 383° and 419° F. It is frequently adulterated, chiefly with alcohol, but occasionally with oil of bergamot. The pure oil fulminates quickly and violently with iodine, and sulphuric acid turns it reddish-brown, the reaction being accompanied by strong thickening. An admixture of alcohol is readily detected by treatment with a small quantity of tannin. If the latter is not altered, the oil contains no alcohol; if, however, it becomes viscid and sticky, it is adulterated. Adulteration with oil of turpentine is detected by treatment with strong alcohol. For the complete solution of 1 part of oil of lavender 5 parts of 90 per cent. alcohol are required; if, however, the oil contains turpentine, the fluid is turbid.

Another kind of oil of lavender, known as *foreign oil of lavender*, to distinguish it from the English oil, or as *spike oil*, is chiefly obtained by distillation from *Lavandula spica* and *L. stoechas*, or Alpine lavenders. This oil, though very good in itself, cannot compare with fine genuine English oil, and brings only about one-tenth of its price. It has a dark green color and slight lavender odor; it has an acid reaction, a specific gravity of 0.9081 at 59° F., and begins to boil at from 338° to 347° F.

*Lemon oil* is obtained by various processes from the fresh peel of the fruit of the *Citrus limonum* Risso. When pure it is almost colorless or bright yellow, very mobile, and has the odor of the fruit. Its specific gravity ranges from 0.8752 to 0.8785; it boils at 148° F., and is miscible in all proportions with absolute alcohol and glacial acetic acid. It is frequently adulterated with turpentine. This may be detected by slowly heating the oil in a dry test-tube with a small piece of copper butyrate to about 338° F., taking care that the temperature

does not exceed 356° F. The copper salt will dissolve in pure oil of lemon with a green color, while in the presence of oil of turpentine a yellow turbid mixture is obtained, reddish-yellow cuprous oxide being separated. This test is also applicable to oils of bergamot and orange-peel. Adulteration with turpentine may also be detected by rubbing a few drops of the suspected sample of lemon oil between the hand, when the turpentine can be distinctly smelled.

By exposure to air and light lemon oil quickly deteriorates, it loses its color, and a thick brown sediment separates out, the specific gravity rises, and the solubility of the oil in 90 per cent. alcohol increases. The oil should therefore be stored in well-stoppered bottles filled up to the neck, in a cool, dark place.

*Lemon-grass oil* is obtained by distillation from *Andropogon citratus*, a hard, coarse grass indigenous to the islands west of the East Indies. The oil is rather viscid, has a greenish to brownish color, and a balm or lemon-like odor. Its specific gravity is 0.899 to 0.903. It dissolves with ease in alcohol, even when diluted, as it gives a clear solution with two or more parts of 70 per cent. alcohol.

Lemon-grass oil is adulterated with fatty oils, for instance, castor oil, and also turpentine. Adulteration with castor oil or other fatty oils is indicated by incomplete solution in 70 per cent. alcohol; the presence of turpentine is best recognized by the smell.

*Linaloe oil*.—This name is used to designate oils from the woods of entirely different trees, namely, a tree indigenous to French Guiana, named by the natives *Likari*, and one in Mexico, *Bursera Delpechiana* Pois. The Mexican oil is a limpid, colorless liquid of an agreeable odor reminding one of lemon oil, so that the mixture of the oil with a little rose oil may pass as an excellent imitation of the oil from citrus flowers. The oil is quite constant in the air. Its specific gravity ranges from 0.870 to 0.880. It gives a clear solution with two or more parts of 70 per cent. alcohol. The oil is frequently adulter-

ated with fatty oils, which can be detected by their insolubility in 70 per cent. alcohol and by the raising of the specific gravity.

*Marjoram oil* is produced by distilling the flowery tops of the sweet marjoram, *Origanum marjorana*. When freshly prepared it is yellowish, but becomes brown by age. It has a pungent smell and a hot, peppery and slightly bitter taste. Its specific gravity is 0.89 to 0.911; when distilled it begins to boil at 365° F., but the temperature rises rapidly to 392° F., and remains constant at between 419° and 428° F., a resinous mass being left in the retort. The oil gives a clear solution with an equal volume of 90 per cent. alcohol.

*Orange oil* or *orange-peel oil* is obtained from the peels of the sweet and bitter orange in a manner similar to that of lemon oil. The oil obtained from the peel of the bitter orange, the fruit of *Citrus bigardia* Duhamel, is chiefly used in the manufacture of liqueurs, whilst for perfumery purposes the oil from the peel of the sweet orange, the fruit of *Citrus aurantium* Risso, is, as a rule, employed. Sweet orange oil has a golden yellow to dark yellow, almost brown color. When rectified it is almost colorless, but does not remain so long. It has a refreshing orange smell and a mild, aromatic taste. Its specific gravity ranges from 0.848 to 0.852; it boils at about 302° F. It is frequently adulterated with both alcohol and inferior oil of other species of citrus. The latter sophistication may be detected by the different behavior of the oils toward alcohol. Genuine orange oil dissolves only by repeated shaking with 12 parts of its quantity of alcohol, while inferior citrus oils require only 6 parts. If, therefore, 7 to 9 parts of alcohol are required for the solution of the suspected oil, adulteration is proved.

*Orange flower oil* is obtained by subjecting the freshly-gathered flowers of the bitter orange to distillation, whilst the flowers of the sweet orange yield an oil not nearly so fine, which is known as *oil of neroli*. Freshly prepared orange flower oil is almost colorless, but on exposure to light turns

brown-red. It has a very agreeable odor of orange blossoms and an aromatic, slightly bitterish taste. The specific gravity of the genuine oil is 0.885 to 0.887. It has a neutral reaction, and is very sparingly soluble in water, but even the smallest possible quantity dissolved suffices to impart to the water the agreeable odor of the oil. In alcohol it is readily soluble. By mixing the clear alcoholic solution with an excess of 90 per cent. alcohol, the mixture becomes turbid and after standing for some time a stearoptene is separated. On account of its high price orange flower oil is frequently adulterated, the most insidious adulterants being bergamot oil and petit-grain oil. The presence of small quantities of these oils cannot be proved, but large additions raise the specific gravity.

*Palmarosa oil*, also known as *Indian verbena oil* and *Indian or Turkish geranium oil*, is obtained from *Andropogon Schænanthus* L. It is colorless to bright yellow and has an agreeable aromatic odor, reminding one somewhat of rose oil, but by no means so strong. It has a pungent but agreeable lemon-like taste. Its specific gravity ranges from 0.888 to 0.896. It dissolves to a clear solution in three or more parts of 70 per cent. alcohol. It is frequently adulterated with castor oil and turpentine, and the Indian product generally with turpentine as well as with alcohol and fixed oils. Such adulterations can be detected by the oil being insoluble in 70 per cent. alcohol.

*Patchouli oil* is obtained from the leaves of the patchouli plant, *Pogostemon patchouli* Pellet, which is chiefly cultivated in the Strait settlements. The oil is produced by submitting the leaves to distillation, they having been previously dried in the shade on bamboo racks. Patchouli oil is a yellowish green to olive-brown, very thick liquid from which, upon standing, crystals sometimes separate out. The odor of the oil is penetrating, somewhat musty and, in an undiluted state, disagreeable, it becoming agreeable only with sufficient dilution, or when mixed with other odoriferous substances. The specific gravity of the pure oil ranges between 0.97 and 0.99. It dissolves completely in an equal volume of 90 per cent. alcohol.

So far as known, patchouli oil is chiefly adulterated with cedar-wood oil, which can be detected by a clear solution not being obtained with an equal volume of 90 per cent. alcohol.

*Peppermint oil* is obtained from the herb *Mentha piperita* L. and some lower species. In commerce are found German, English, American and Japanese peppermint oils. For perfumery purposes the American and German oils are as suitable as the English oil, which is considered a superior product possessing a very fine, pure taste and is chiefly in demand by the manufacturers of liqueurs. The Japanese oil has a peculiar, rather greasy smell and taste, and is not much sought after.

Crude peppermint oil is of a greenish to yellow color and contains slimy substances. When rectified by repeated distillation with water it is colorless, limpid, has a penetrating odor of peppermint and a pungent taste, imparting at the same time a sensation of coldness to the tongue and palate. Old oil, as well as Japanese oil, has a disagreeable taste. In Japan enormous quantities of peppermint oil are produced, but on account of this disagreeable bitter taste the oil is only suitable for perfuming soaps and the production of cheap toilet articles. It is also used for obtaining menthol. It is brought into commerce as "separated" or "unseparated" oil or in crystals, *i. e.*, either only the liquid portion of the oil is sold, or only the solid portion, or a mixture of both, the latter containing from 30 to 50 per cent. menthol crystals.

The various peppermint oils do not show any great difference in respect to specific gravity, but the variation is nevertheless sufficiently wide to render this property indicative of the origin of the oil. The specific gravity of American oil is generally 0.910 to 0.920, of English oil 0.900 to 0.910, and of normal Japanese oil 0.895 to 0.900, while that of the liquid oil remaining from the production of menthol is 0.895 to 0.905.

With 3 to 5 times its volume of 70 per cent. alcohol at 68° F., English peppermint oil gives a clear solution. If more alcohol be added, the solution generally remains clear, but

sometimes begins to show slight opalescence which, however should not reach the point at which oil drops begin to separate. The solubility of Japanese oil, from which menthol has been extracted is, as a rule; the same as with English oil, but often somewhat slighter. American oil is never quite soluble in 70 per cent. alcohol, but always leaves a certain proportion of insoluble parts which separate from the rest as a layer upon the surface. Even in 90 per cent. alcohol American oil is not always soluble in all proportions. To attain perfect solubility at 68° F.,  $\frac{1}{2}$  part by volume of alcohol is generally required.

When 5 drops of American or English peppermint oil are mixed with 1 cubic centimeter of glacial acetic acid, a blue color begins to show itself after a few hours. This gradually acquires greater intensity, the maximum being reached in about 24 hours. At that time American oil shows a very deep blue color with magnificent copper-colored fluorescence. In the case of English oil the coloration is much weaker, often only reaching a pale blue color with faintly reddish fluorescence. Japanese oil alone does not give these reactions, the mixture remaining quite colorless.

Gentle heat or immersion of the test-tube in boiling water considerably accelerates the reaction. When this is done, however, a much less pure blue color is obtained, the tint in that case being more violet. Free access of air is absolutely necessary for the success of the reaction. If air is carefully excluded no coloration occurs even with American oil, after several days.

Peppermint oil is most frequently adulterated with turpentine which can be detected by the oil not being perfectly soluble in 90 per cent. alcohol.

*Petit grain oil* is obtained by distilling the unripe fruits, flowers, leaves, young shoots, etc., of different species of citrus, the best quality of oil being produced from the leaves of the bitter orange. The smell of petit-grain oil, while resembling that of orange flowers, is by no means so fine. Its color is

yellowish, and its taste similar to that of orange peel oil and quite mild. Its specific gravity ranges between 0.887 and 0.900. It gives a clear solution with 2 parts 80 per cent. alcohol. It is adulterated with orange oil, lemon oil and turpentine, such adulterations being detected by the lowering of the specific gravity.

*Rose oil.*—This precious oil is obtained from various species of roses. The principal commercial source of rose oil is a circumscribed patch of ancient Thrace or modern Bulgaria, where the variety used for distilling purposes is the so-called Thracian rose, a plant of exceedingly rapid growth. The average quantity of product is estimated by Baur at 0.037 to 0.040 per cent.; another authority says that 3200 pounds of roses give 1 pound of oil.

Pure rose oil, carefully distilled, is at first colorless but quickly becomes yellowish; its specific gravity is 0.870 at 72.5° F., its boiling point is 444° F., it solidifies at 52° to 61° F., or still higher, it is soluble in absolute alcohol and in acetic acid. The most usual and reliable tests of the quality of rose oil are: 1, its odor; 2, its congealing point; and 3, its crystallization. The odor can be judged only after long experience. That of the concentrated oil is intense, penetrating and diffusive, and to most persons unpleasant, the fine agreeable odor being only brought out by dilution. A good oil should congeal in five minutes at a temperature of 54.5° F., fraudulent additions lowering the congealing point. The crystals of rose-stearoptene are light, feathery, shining plates, and fill the whole liquid.

It is evident that such a valuable oil as rose oil is very much exposed to adulteration. Indeed, it is said that only adulterated oil reaches the market, the sophistication, chiefly with geranium and ginger-grass oils, taking place at the home of the oil. The general characters of these oils are so similar to those of rose oil, even the odor bearing a distant resemblance, that detection is very difficult. Greiner recommends the following as a reliable test: Put one drop of the suspected

oil in a dry test-tube and add four drops of concentrated sulphuric acid. A perceptible rise in the temperature takes place, and the mixture must be allowed to stand until it becomes cool. Two grammes of absolute alcohol are then to be added and the mixture well shaken. With pure oil the mixture will be slightly opalescent, and, on heating, will turn yellowish brown, the color remaining on cooling the solution. In the presence of ginger-grass or geranium oil, the solution will be turbid and an insoluble precipitate soon forms. Pure rose oil retains its characteristic odor when subjected to this test, but the mixture with the other oils evolves unpleasant odors.

When fatty oils, such as sesame, almond, etc., are used as adulterants, the usual test, made by placing a drop on white paper and heating over an alcohol flame, shows their presence in the greasy stain which remains. Pure rose oil is entirely volatile.

The presence of spermaceti, which is usually used for artificially heightening the apparent proportion of stearoptene, is easily recognizable from its liability to settle down in a solid cake, and from its melting at 122° F., whereas the stearoptene fuses at 81.5° F.

*Rosemary oil* is obtained by distillation from the rosemary, *Rosmarinus officinalis*. It is colorless to pale green, very limpid, and of a more aromatic than sweet odor characteristic of the plant. Its specific gravity is 0.90 to 0.915 at 59° F.; it boils at from 331° to 334° F., and congeals at from 81° to 86° F. It is frequently adulterated with turpentine; the sophistication can only be detected by the difference of solubility in alcohol. Pure rosemary oil dissolves in an equal volume of 90 per cent. alcohol, while oil adulterated with turpentine requires a far greater quantity for complete solution.

*Sassafras oil* is obtained by distillation from the wood, bark and root of the sassafras laurel, indigenous to the southern parts of North America. The oil, when first distilled, is colorless or pale yellow, but it becomes reddish with age. Its taste

is pungent and aromatic, being agreeable to most persons. Its specific gravity ranges from 1.07 to 1.09, increasing somewhat by age. It is readily soluble in alcohol, ether, chloroform, and miscible with other fixed and volatile oils. The crude oil is composed of about 90 per cent. saffrol held in solution by 10 per cent. of saffrene.

Sassafras oil, according to Gildermeister and Hoffman, is very frequently adulterated with camphor oil. As all the constituents of sassafras oil are contained in camphor oil, its detection is very difficult, and it can only be done with certainty when the physical properties, especially the specific gravity, show marked variations. Fractions of camphor oil of a specific gravity corresponding to that of sassafras oil are sold as artificial sassafras oil.

*Thyme oil* is obtained by distillation from the flowering herb *Thymus vulgaris* L., a plant indigenous to Southern Europe, and elsewhere largely cultivated in gardens. In a crude state the oil is greenish yellow to red, when rectified, colorless, but becomes yellow to brown-red by standing. Its odor is strong and agreeable like that of the plant, and its taste, as a rule camphor-like, cooling and somewhat pungent. Its specific gravity ranges from 0.87 to 0.904 at 59° F., the oil from fresh thyme possessing a higher specific gravity than that from the dried herb. Thyme oil is soluble in half its volume of 90 per cent. alcohol and 1 to 2 parts of 80 per cent. alcohol, but generally 15 to 30 parts of 70 per cent. alcohol are required for a clear solution. The constituent on which the value of thyme oil depends is thymol, the withdrawal of which constitutes the principal sophistication of the oil. It is also adulterated with turpentine as well as with petroleum.

*Vetiver oil* is obtained from the rhizome of *Andropogon muri-catus* Retz. The oil is obtained by distillation either from the fresh root in India, but chiefly in England and Germany from the dried root known as *cus-cus*. It is viscid, of a red-brown color and has an intense odor very much like that of orris oil. At 59° F. it is heavier than water, but lighter at a higher tem-

perature. It is chiefly adulterated with fatty oils, which can be detected by the solubility in alcohol; with 2 parts of 80 per cent. alcohol the oil should give a clear solution.

*Wintergreen oil* is obtained by distillation from the herb *Gaultheria procumbens* L., a plant common to North America. An oil said to be identical with the ordinary wintergreen oil is also obtained from the leaves of *Gaultheria punctata* and *G. leucocarpa*, which grow especially on the summits of extinct volcanoes in Java. Wintergreen oil is a thick, yellowish or reddish liquid with a penetrating, stupefying odor which, however, becomes agreeable with sufficient dilution; its taste is sweet and at the same time warm aromatic. It is almost insoluble in water, but imparts to it its peculiar odor and taste; it dissolves readily in alcohol, ether, chloroform, etc. Its specific gravity is 1.18 at 59° F.

An oil very similar to wintergreen oil and frequently sold as such is obtained by distillation with water from *Betula lenta*, an American species of birch. It differs somewhat in smell from genuine wintergreen oil, but cannot be distinguished from *methyl salicylate* or *artificial wintergreen oil*. The latter has for a number of years been brought into commerce and is largely used for perfuming purposes. It is a colorless liquid, possessing the odor of the natural oil and a specific gravity of 1.176. On shaking a few drops of the artificial product with water, the oil becomes turbid while natural oil immediately separates clear.

*Ylang-ylang oil* is obtained by distilling the flowers of *Cananga odorata*, Hooker, which grows in the Philippines, the Straits of Malacca and the Indian Archipelago, and is largely cultivated in South Asia. The oil which is chiefly brought into commerce from Manila, is colorless or slightly yellowish and possesses an exquisite odor, somewhat partaking of the jasmine and the lilac. Its specific gravity is 0.980 at 59° F. It is soluble with difficulty in alcohol; with one half to two volumes of 90 per cent. alcohol it generally gives a clear solution which, on the further addition of alcohol, as a rule,

becomes turbid. With chloride of iron an alcoholic solution of the oil gives a violet coloration.

An inferior quality of ylang-ylang oil is known as *Cananga oil*. It is derived from the same plant, the difference in quality being due to less care in the preparation. Cananga oil does not completely dissolve in 90 per cent. alcohol, 1½ to 2 volumes of 95 per cent. alcohol being generally required for solution. On further addition of alcohol the solution becomes turbid, but from the resulting opalescent liquid no drops separate out even on long standing.

*Resins and balsams*.—The term *resins* is applied to certain organic substances which are closely related to volatile oils, in so far as many of them are formed from the latter by oxidation. In nature most resins also occur mixed with essential oils.

The resins are generally divided into *hard resins*, *soft resins* or *balsams*, and *gum resins*. The hard resins are, at the ordinary temperature, solid, hard and brittle, can be readily pulverized, and contain little or no volatile oil. The soft resins or balsams are kneadable, and sometimes even semi-liquid; they represent solutions of resins in essential oils, or a mixture of essential oil and resin. On exposure to the air they are gradually changed by the essential oil suffering oxidation, they becoming more or less hard, and may be converted into actual resins. The gum resins are mixtures of vegetable gum, resin and essential oils, and are obtained by inspissation of the milky juice of several plants. When triturated with water, they yield a milky, turbid fluid, and dissolve only partially in alcohol.

Of the hard resins, benzoin alone is used in perfumery; of the balsams, Peru balsam, tolu balsam and storax; and of the gum resins, myrrh and opopanax.

*Benzoin* is obtained from *Styrax benzoin* Dryand, a tree which grows in Java, Sumatra and Siam. According to the appearance of the product three varieties are distinguished: Benzoin in tears, benzoin in almonds, and block benzoin. The *benzoin*

*In tears* forms loose, smoothly terminated, longish masses of a homogeneous appearance, of an opalescent luster, and at first of a whitish, and later on of a yellowish, reddish or brownish color. The separate pieces are up to 3 millimeters in diameter, though the Siam benzoin in tears frequently consists of still larger and generally decidedly flattened pieces.

*Benzoin in almonds* consists chiefly of white pieces, becoming later on brownish ; they have a waxy luster and are imbedded in a lustrous brown-red resinous mass. *Block* or *ordinary benzoin* has also an amygdaloid structure, but is not so rich in almonds as the preceding variety, and possesses either a fine granular or colophony-like ground-mass, and is frequently contaminated with parts of plants. The outside of the variety occurring in commerce in large lumps shows, according to the mode of packing, the imprint of leaves or of coarse packing cloth.

All varieties of benzoin possess a peculiar odor, that of the better varieties being agreeable, and have a sweet, aromatic but pungent taste. The melting-point generally lies between 176° to 203° F., that of the tears and the almonds being lower than that of the ground-mass. A low melting-point is accepted as a mark of quality, Siam benzoin, which is considered the best, melting at 167° F. Some varieties, for instance, those brought from Singapore, have a pronounced vanilla-like odor ; the presence of vanillin has been established in them.

Benzoin is sparingly soluble in chloroform, only partially so in ether, and completely in alcohol. Benzine withdraws only benzoic acid from the dry, powdered benzoin. All varieties of benzoin dissolve in concentrated sulphuric acid to a fluid of a beautiful color, from which benzoic acid, if present, separates out in crystals by the gradual addition of water.

*Peruvian balsam* comes from the Balsam Coast, San Salvador, Central America, where in the mountain forests back of the coast grows the balsam tree, *Myroxylon pereira* Klotzsh. The tree begins to yield balsam when it has become five years old, the collecting time commencing in the dry season in the first

days of November. The trunks of the trees are belabored with hammers so that the bark is detached in strips. After a few days the bark thus loosened is burnt off by means of torches, whereupon a balsamic fluid oozes from the young wood, which is absorbed by pieces of cloth or rags placed upon the denuded places. When the rags are thoroughly saturated with balsam they are squeezed out and then thrown into an earthen pot filled with boiling water, whereby the balsam is detached and collects on the bottom of the vessel. By this process *Balsamo de Trapo* is obtained. By boiling the bark which falls off, a small quantity of a poorer quality of balsam called *tacuasonte* is obtained.

Crude Peruvian balsam is a gray-green to dirty yellow fluid of the consistency of syrup.

Ordinary (black) Peruvian balsam is a black-brown fluid, transparent and dark (honey-yellow in thin layers), which retains its consistency even after being kept for years and deposits no crystals. It shows a slight acid reaction, has an agreeable odor reminding one of gum benzoin and vanilla, and at first a mild, but, later on, a sharp and pungent taste.

The specific gravity of pure Peru balsam formerly ranged from 1.14 to 1.16, but at present from 1.135 to 1.145, this change in the specific gravity being very likely due to a different process of purification.

Peruvian balsam is miscible in all proportions with absolute alcohol and is almost completely soluble in 90 per cent. alcohol. In fatty oils it is partially soluble. Benzine dissolves from it only the nearly colorless cinnamein, of which it contains up to 45 per cent.

As adulterants, are used : Alcohol, volatile oils, fat oil, especially castor oil ; further, copaiba balsam, Canada balsam, gurjun balsam, storax, benzoin, and asphaltum. The establishment of these adulterants is connected with difficulties ; but the properties of Peru balsam are so characteristic that it is quite easy to detect whether it is genuine and pure, or not, the specific gravity and proportions of solubility deserving

especial attention in this respect. The test by the specific gravity is available, since most of the adulterants render the balsam specifically lighter, especially alcohol, but also copaiba balsam (specific gravity, 0.95), castor oil (0.96), oil of turpentine (0.87), gurjun (0.96), etc. The customary procedure is as follows: Prepare a common salt solution of 1.25 specific gravity, by dissolving 1 part of dried sodium chloride in 5 parts of distilled water; drop the balsam into the solution; every drop of pure Peru balsam sinks in a roundish form to the bottom, but if the drop again comes to the surface and spreads out upon it, it is a sure sign of some kind of adulteration. However, the change in the specific gravity by the admixture of fat oils is but very slight, since the balsam can only be mixed with them to a conformable fluid in the proportion of from 7 to 10 to 1. Castor oil forms an exception in this respect, it being miscible also in other proportions.

Petroleum-ether is an excellent testing agent. Bring into a test-tube about 2.5 grammes of Peru balsam, and 6 to 7 centimeters of petroleum-ether, close the tube with the finger and shake vigorously; a brown, thickly-fluid mass adheres in unequal layers to the sides of the tube, and before running together remains in this position one to two minutes after the petroleum-ether has been poured into a porcelain saucer. If, however, the mass is thinly fluid, and does not, in the above-mentioned manner, adhere to the sides of the tube, but, after shaking, collects below the petroleum-ether, the balsam is adulterated. After shaking, immediately pour off the petroleum-ether; if the latter is almost colorless, or but slightly colored yellowish, the balsam is pure; if, however, it is turbid, and soon forms a sediment, or if it is yellow or brownish, or brown, the balsam is adulterated.

*Tolu balsam* is the produce of *Myroxylon toluiferum*, Humb., Bonpl. et Kunth, *Toluifera balsama*, L., a tree growing in Northwestern South America. It exudes during the heat of the day, and is collected in gourds. It soon hardens, by which it is distinguished from Peruvian balsam. In commerce

two kinds of Tolu balsam are found, one of the consistency of turpentine, and the other solid. The first kind, Brazilian balsam, forms a semi-liquid, turpentine-like, sticky mass of the color of copaiba balsam. By long storage it becomes hard and brownish. The second variety, Tolu or Carthagena balsam, is a brittle, more or less translucent yellow-brown or reddish-brown resin of a granular or crystalline appearance. It softens at about 86° F., and melts between 140° and 149° F. Viewed under the microscope it appears rich in crystals of separated cinnamic and benzoic acids. Its specific gravity varies between 1 and 2. Both varieties of Tolu balsam have an aromatic, slightly pungent and sourish taste, resembling somewhat that of Peruvian balsam. They are readily soluble in ordinary spirits of wine, alcohol, acetone, chloroform and potash lye, but insoluble in petroleum-ether and carbon disulphide.

Commercial Tolu balsam is frequently more or less mixed with vegetable remains which, however can be readily detected with the microscope, especially after the solution of the resinous constituents. It is frequently adulterated with turpentine or pine rosin. Such adulterations may be detected by carbon disulphide, which completely dissolves these substances, but not the Tolu balsam. When pure Tolu balsam is triturated with concentrated sulphuric acid, a cherry-red liquid is obtained, which does not evolve sulphurous acid, as is the case in the presence of turpentine-rosins.

*Storax* comes from *Liquidambar orientale*, Mill., a plantain-like tree, which forms handsome, dense forests in Southern Asia Minor, especially in Cyprus.

Liquid storax is a sticky, opaque substance of the consistency of turpentine. It has a mouse-gray color, which by contact with the air becomes brown on the surface, an agreeable benzoin-like odor, and a sharp, pungent, aromatic taste. It is heavier than water, its specific gravity being 1.112 to 1.115. On losing its content of moisture (by drying out when heated) it becomes brown and clear. When exposed to the air in a

thick layer it does not completely dry, and in a thin layer only after considerable time; but when pressed with the finger always shows a certain stickiness. It is partially soluble in spirits of wine, yielding with it a more or less turbid solution. It is also incompletely soluble in oil of turpentine, benzine, petroleum-ether, and chloroform. Viewed under the microscope liquid storax appears as a colorless thickish fluid, intermingled with larger and smaller drops, fragments of bark tissue, and now and then, perhaps, with crystals of styracic acid and cinnamic acid.

Good liquid storax should yield to 90 per cent. alcohol at least so much soluble matter that the dried residue of the filtered alcoholic solution amounts to 65 per cent. of the quantity of storax.

Ordinary liquid storax, which has not been stored too long, contains 10 to 20 per cent. of water and about the same quantity of impurities (fragments of plant tissue) which remain behind on treatment with the above-mentioned solvents.

Liquid storax is said to be adulterated with the turpentines of some species of larch and pine. Such adulteration is primarily detected, according to Hager, by the specific gravity. Take up a drop of the balsam with a knitting-needle, and by heating the needle make it fall into a cold solution of 1 part common salt and 8 parts water. On stirring, the drop must sink, otherwise adulteration with turpentine is very likely. Next bring 5 grammes of the storax into a test-tube, melt it in the water-bath, add  $\frac{1}{2}$  volume of absolute alcohol, and mix by shaking; then compound the mixture with several times its volume of petroleum-ether, shake vigorously, allow to settle, and decant the layer of petroleum-ether. Repeat twice this shaking with petroleum-ether; then evaporate the petroleum-ether solution in a tared flask on the water-bath. The residue remaining after evaporation is colorless, bluish opalescent, and of an agreeable odor; in the presence of turpentine it is yellowish and has the, not to be mistaken, odor of turpentine.

*Myrrh* is a gum-resin, which comes from several kinds of the species *Commiphora* indigenous to South Asia and Abyssinia. The gum-resin exudes spontaneously as a white, oil-like mass and, after hardening, is collected by the natives and brought chiefly to Berbera, a small seaport opposite Aden, to be exchanged for English and Indian goods. From there, by way of Aden and Bombay, it reaches the European market. In Bombay the first sorting takes place, which is, however, superficial, and hence has to be repeated in Europe (London). According to Parker, ten different resins are admixed with myrrh, especially bdelium resins.

In commerce *Myrrha electa* and *Myrrha vulgaris* or *in sortis* are distinguished. *Myrrha electa*, the best quality, occurs in pieces of irregular form and variable sizes, consisting of tears—either distinct or agglomerated—usually covered with a fine powder or dust. The surface is seldom smooth, but generally rough or granular. The color varies, being pale reddish, yellow, red or reddish-brown. The fracture is conchoidal, seldom smooth, but rather granular, rough, of a fatty luster, and sometimes shows whitish striae or veins, or opalesces like flint. The fractured edges are more or less translucent; thin disks or splinters are translucent or transparent. The specific gravity is, according to Hager, 1.195 to 1.205, and according to Ruickholdt, 1.12 to 1.18. A *Myrrha electa* is the better, the more fragile, friable and paler in color it is, and the more rapidly it ignites and burns with a yellow, sooty flame. Poorer qualities may be recognized by the dark brown color and dirty appearance. Myrrh is with difficulty rubbed to a fine powder, this being possible only after drying, which must, however, be done at a very moderate heat to prevent loss of essential oil.

Myrrh, according to Hager, consists in 100 parts of about 2.5 parts essential oil (myrrhol), 25 to 35 parts resin (myrrhin), 55 to 65 parts gum soluble in water, 3 to 8 parts salts, impurities and water. Water forms with myrrh an emulsion and dissolves the gum. The resinous constituents are dissolved by spirit of wine.

Myrrh is frequently contaminated with bark, which forms either a film of cork as thick as paper or a crust of a fibrous and, at the same time, brittle nature. Sand or small pebbles are also frequently mixed with the myrrh. Other varieties of gum or gum-resin, which considerably decrease the value of the product, are often found in the commercial article, the inferior qualities especially being adulterated and mixed with dark pieces of Suakim gum, gum of the plum or cherry tree, bdellium, and similar substances, which are partially moistened with myrrh tincture, and scattered over with myrrh powder. Adulteration with gum-arabic, gum of the plum or cherry tree, which are coated with alcoholic myrrh solution, is recognized by the paler luster, greater transparency, and mucilaginous taste. Pieces of resin melt on heating, while myrrh only swells up. Bdellium is detected by the dark or black-brown color, toughness, less bitter taste, and by crackling and spitting when held in the flame of a candle, as well as by the reaction of myrrh with nitric acid discovered by Bonastre. By mixing 5 cubic centimeters of alcoholic myrrh tincture with 5 to 10 drops of fuming nitric acid, a rose-color coloration, passing into red, results. Parker gives the following method for testing myrrh: Prepare a tincture of 1 part myrrh and 6 parts spirit of wine. Saturate with this tincture white filtering paper, allow it to drain off, and then wrap it around a glass rod moistened with nitric acid of 1.42 specific gravity. With genuine myrrh the paper immediately becomes deep yellow-brown and then black, while the edges of the paper strip appear dark purple red. When a few drops of the tincture of myrrh are allowed to dry in, a transparent residue remains behind. The tinctures of spurious articles, with the exception of bissabol, give turbid residues.

*Opopanax*.—Various gum resins are known under this name, but that of interest to the perfumer comes from the *Balsamodendron Kaful* Kunth, indigenous to Persia. It forms grains or lumps of a red yellow or brown color and has a fracture of a waxy luster. It can be rubbed to gold-yellow powder. It

has a strong and peculiar odor, and a very bitter and balsamic taste. With water it forms an emulsion, while it is only partially soluble in spirit of wine. It contains very little essential oil, and a resin which melts at 212° F., and is soluble in ether and aqueous alkalies. It further contains gum, organic and inorganic salts and foreign admixtures.

*Perfumes of Animal Origin.*—*Ambergris*.—This is an odorous solid substance found floating on the sea in tropical climates, and in the cæcum of the cachet or spermaceti whale (*Physeter macrocephalus*). It has been supposed by some to be a morbid secretion of the liver and intestines analogous to biliary calculi; but, according to Mr. Beale, it consists of the mere indurated feces of the animal, perhaps somewhat altered by disease. The color of ambergris is grayish-white and yellow marbled. It has a pleasant musk-like odor, which is heightened by warming, the odor being peculiar, and not easily described or imitated. It does not effervesce with acids; melts at 140° to 150° F. to a yellowish resin-like mass, and at 212° F. flies off as a white vapor. It dissolves easily in absolute alcohol, ether, and also in fat and volatile oils. A factitious ambergris is said to be made as follows: Orris powder, spearmaceti, gum benzoin, of each 1 pound, asphaltum 3 to 4 ounces, ambergris 6 ounces, grain musk 3 drachms, oil of cloves 1 drachm, oil of rhodium  $\frac{1}{2}$  drachm, liquor ammoniae 1 fluidounce. Beat to a smooth, hard mass with mucilage, and make into lumps whilst soft.

*Civet*.—Under this name is known an animal secretion which originates from *Viverra zibetha*, the Asiatic, and *Viverra civetta*, the African civet. It separates in these animals from particular glands into a sort of pocket which is situated between the anus and the genital organs and opening outside. The wild animal squirts this mass from time to time spontaneously; from the animals kept in captivity it is taken with a spoon. Civet forms a smeary, soft, at first white, after a while brownish mass, becoming in time more consistent. It has a peculiar musk or ambergris-like fragrance and a dis-

agreeable, bitter, irritating taste. It melts when heated, puffs up, takes fire, and burns with a bright flame.

*Musk* is a peculiar concrete substance obtained from *Moschus moschiferus*, an animal bearing a close resemblance to the deer in shape and size, and indigenous to the high plateaus of Asia. The musk is contained in an oval, hairy, projecting sac, found only in the male, situated between the umbilicus and the prepuce. It is from two to three inches long and from one to two broad. In the vigorous male animal the sac contains sometimes 6 drachms of musk, but in the old, seldom more than 2 drachms, and none in the young.

Musk is in grains or lumps concreted together, soft and unctuous to the touch, and of a reddish-brown color resembling that of dried blood. The odor is strong, penetrating, and so diffusive that one part of musk communicates its smell to more than 3000 parts of inodorous powder. The taste is bitter, disagreeable and somewhat acrid.

Musk is very much adulterated, very little of the genuine article reaching the market. The Chinese are adepts in this sophistication. Dried blood, on account of its resemblance to musk, is among the most common adulterations, but besides this, sand, iron filings, hair, the dung of birds, wax, asphalt and many other substances are introduced. They are mixed with a small portion of musk, the powerful odor of which is communicated to the entire mass and renders the discovery of the fraud sometimes difficult. The bag containing the musk should have the characters before described as belonging to the natural sac and present no sign of having been opened. Genuine musk burns with a white flame and leaves a white, spongy charcoal; that which burns with difficulty, has a feeble odor, and a color either pale or entirely black, feels gritty to the finger, is very moist so as to lose much weight in drying, should be rejected.

*Artificial Perfumes.*—In speaking of the essential oils used in perfumery, attention has been drawn to the fact that, thanks to the progress in chemistry, there are now at the disposal of

the soapmaker and perfumer quite a number of artificial perfumes. These products are partly bodies separated from essential oils and partly bodies obtained by synthesis. It is not within the province of this work to enter into the process of manufacture of these products, but the more important ones may be briefly referred to.

*Anisic aldehyde* is fluid at the ordinary temperature. Its smell reminds one of that of the flowering hawthorn. It is found in commerce under the name of *aubepine* and is quite extensively used for soaps, it being especially useful in combination with orange oil, petitgrain and similar essential oils.

*Citral* forms a thin, faintly yellowish oil, with a penetrating smell of lemons and a sharp burning taste.

*Cumarin* forms small colorless crystals of a silky luster. It is very hard, cracks between the teeth, shows a smooth fracture, and sinks in water. It has a very agreeable aromatic odor which, on rubbing the substance with the fingers, becomes like that of oil of bitter almonds. It has a bitter, warm and pungent taste.

*Geraniol* forms, in a pure state, a colorless liquid, with a specific gravity of 0.882 to 0.885, and is easily soluble in alcohol.

*Heliotropin* or *piperonal* forms small colorless prismatic crystals, which have an agreeable odor of heliotrope. Heliotropin melts at about 104° F., and volatilizes at a higher temperature without leaving a residue. It is soluble in alcohol and ether, and insoluble in cold water; in hot water it melts to an oily liquid, which floats upon the water.

*Hyacinth* consists chiefly of terpinol. It is easily soluble in all liquids employed in perfumery. It is useful not only for the preparation of hyacinth soaps, but is also to a considerable extent used in combination with rose and geranium oils for rose soaps.

*Linalool* is an almost colorless liquid with a specific gravity of 0.878. It is easily soluble in alcohol and has a remarkably fine odor. It is a substitute for linaloe oil, but is considerably more exhalent.

*Neroli oil*.—An artificial oil of neroli brought into commerce forms fine dazzling white crystalline scales, which melt at 97° F.; it should therefore be stored in a cool place. It dissolves in nearly all proportions in alcohol, fatty and essential oils, and is especially recommended for perfuming cheap toilet soaps, one pound of it being at the utmost required for 200 lbs. of soap. By combining it with linalool, orange oil and cumarin an agreeable and effective perfume is also obtained.

*Safrol* is a colorless or slightly yellowish liquid with a specific gravity of 1.106. It is very useful for covering the unpleasant fatty smell in soaps and for that reason is largely used for perfuming domestic soaps. For soaps from good fats 4 to 8 ozs. are sufficient for 200 lbs. of soap.

Artificial bitter almond oil and artificial wintergreen oil have been referred to under the respective natural oils.

*Tinctures*.—These are prepared from the resins and balsams previously described, as well as from the perfume-substances derived from the animal kingdom. Most of these substances, if not already found in commerce in the form of powder, are, previous to extraction, pulverized, or at least comminuted as much as possible. The infusions should be stored in a moderately warm room, and vigorously shaken several times every day. When extraction is finished the product is filtered through paper. The substances to be used should be fresh and genuine, and the alcohol free from fusel oil, since a perfect tincture can only be obtained under these conditions. It is advisable to have always a sufficient supply of tinctures on hand, since their aroma improves by age.

*Musk tincture*.—Tonkin musk 11 drachms, rose water 8 ozs., best quality alcohol 2 quarts.

Carefully empty the musk sac into a glass flask, add the rose water, and let the flask stand for about 10 days, shaking frequently. Then add the alcohol and let the whole stand for several weeks, shaking frequently. Cut up the empty musk sacs into as small pieces as possible, and, in another bottle, treat them in the same manner as their contents; distilled

water may, however, be used instead of rose water. The object of the water is to soften the musk, which swells up, so that the alcohol can better penetrate into the cellular tissue and absorb the aroma.

The extract from the empty musk sacs is used for cheaper products, or mixed with the extract from the contents of the sacs, according to whether a more or less fine quality of tincture is to be obtained. A still higher yield might, perhaps, be obtained by the use of a machine for comminuting the musk, which grinds the sac to atoms, whereby the cellular tissue is still more completely disintegrated than by cutting up.

*Civet tincture.*—Civet 5½ drachms, best quality of alcohol 3 pints.

Civet in its natural state being, with difficulty, soluble in alcohol, triturate it in a mortar to a pulverulent mass together with some dry substance, for instance, whiting or exhausted orris-root powder. The mixture is then brought into a glass flask, the alcohol added, and the whole frequently shaken.

*Ambergris tincture.*—Ambergris 5½ drachms, alcohol of the best quality 1 quart.

Ambergris dissolving readily in alcohol, pulverizing is not required, but if it is done great care should be exercised to prevent loss of this expensive substance. Ambergris is not so much distinguished by its aroma as by its indestructibility, which renders it especially suitable for fixing odors.

*Benzoin tincture.*—Benzoin (Siam) 2 lbs., best quality of alcohol 3 quarts.

Convert the benzoin into a coarse powder, bring it into a flask, add the alcohol and shake thoroughly. Solution takes place in 10 to 12 days.

Siam benzoin is the finest and most expensive and is indispensable for *Extraits d'Odeurs*. For cheaper products of perfumery, Sumatra benzoin answers very well.

*Opopanax tincture.*—Opopanax 2 lbs., best quality of alcohol 4 quarts.

Reduce the opopanax to a coarse powder, bring it into a bottle, add the alcohol and shake frequently.

*Peru-balsam tincture*.—Peru balsam 8 ozs., best quality of alcohol 5 quarts.

*Tolu-balsam tincture*.—Tolu balsam 3 lbs., best quality of alcohol 5 quarts.

Bring the alcohol into a bottle. Tolu balsam cannot be reduced to a powder, hence it is necessary to keep it cold, whereby it becomes brittle so that it can be cut up with a sharp instrument and a hammer. The pieces detached are rapidly brought into the alcohol, solution taking place in about 14 days. If the alcohol were added to the Tolu balsam, the latter would ball together, rendering solution very difficult. Frequent vigorous shaking is necessary.

*Myrrh tincture*.—Myrrh 1 lb., alcohol 2 quarts. The myrrh should previously be coarsely powdered.

*Storax tincture*.—Liquid storax 2 lbs., alcohol 5 quarts.

Pour the alcohol into a bottle. Liquefy the storax by placing the vessel containing it in warm water, and then pour it in a very thin stream into the bottle containing the alcohol.

## CHAPTER XIX.

### TESTING SOAPS.

In testing soaps the following points have to be taken into account: 1. Content of water. 2. Proportion of fatty acid to alkali. 3. Nature of the alkali and the fatty acid, relatively of the rosin. 4. Intentional admixture of organic or inorganic substances.

*Determination of the content of water.*—In the determination of the content of water special care must be had to obtain a fair average sample. The content of water of the outer portions being less than that of the interior, thin cross-sections should be taken from bars of hard soap, and the samples of soft soap from the centre of the barrel. In drying soap containing much water, it frequently happens that it melts when exposed to a temperature of 212° F. and becomes covered with a film which prevents the escape of the aqueous vapor. To prevent melting together, Loewe recommends first to dry 8 to 10 grammes of the soap, previously reduced to fine shavings, at from 140° to 158° F., and then at from 212° to 221° F., until the weight is constant. This is best done upon a large watch-crystal, which for weighing is covered with another one fitting hermetically. For the determination of water, Gladding recommends to weigh a beaker-glass of about 100 cubic centimetres capacity together with a glass-rod. The bottom of the beaker-glass is to be previously covered 1.3 centimetres deep with annealed quartz sand. Then introduce into the beaker-glass about 5 grammes of the soap, weigh again, add about 25 cubic centimetres of alcohol, and heat with occasional stirring over a water-bath until the weight is constant. The loss is water. This method is especially re-

commended for soft soap, which it is difficult to get entirely dry by Loewe's process. The exact determination of water in soap being difficult at the best, as the dried soap readily reabsorbs water, the indirect method, *i. e.*, determining all the other constituents of the soap and calculating the water from the difference, is more accurate. Moreover, with soap that contains free alkali, alcohol, essential oils, or larger quantities of glycerin, determination of the loss of weight by heating is not possible.

*Determination of the content of fatty acid.*—Take a sample of 6 to 10 grammes, partially from the interior of the bar and partially from the exterior, in order to obtain one with the average content of moisture. Place the sample in a porcelain dish, pour 20 to 30 times its weight of diluted sulphuric acid (1 part acid to 12 parts water) over it, and heat until the clear fatty acid floats on the surface. From oil-soap the fatty acid separates with greater ease than from tallow-soap, but, as it does not completely congeal, it is difficult to remove without loss, from the fluid beneath. This is overcome by the addition of an accurately weighed quantity (6 to 10 grammes) of thoroughly dried white wax or stearin, which is melted together with the fatty acid. The mass after cooling represents a coherent, hard cake, which can be readily lifted from the fluid by means of a spatula. The cake is placed upon a filter and washed with distilled water as long as the latter takes up sulphuric acid, *i. e.*, shows the reaction with barium chloride. The cake of fatty acid is then dried under a bell alongside a vessel containing sulphuric acid, preferably but not absolutely necessary, under an air-pump, until the weight remains constant. Then deduct the weight of the stearin or wax that has been added from the total weight of the cake. The remainder, provided the soap under examination contains no rosin, represents the hydrate of fatty acid and, therefore, cannot be set down as the result of the analysis, and a reduction to anhydrous fatty acids has first to be made.

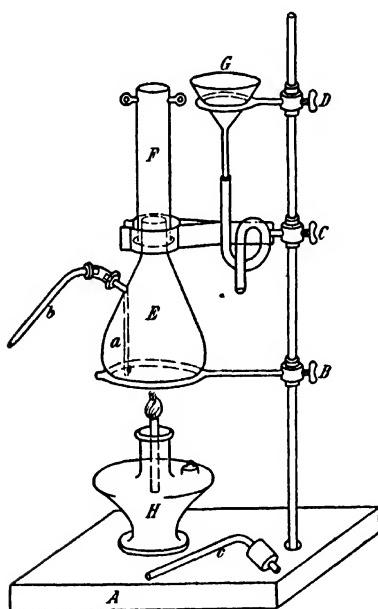
Since 100 parts of stearic acid,  $C_{18}H_{36}O_2$ , correspond to

96.83 parts of stearic acid anhydride,  $(C_{18}H_{36}O)_2O$ , 100 parts of palmitic acid,  $C_{16}H_{32}O_2$ , to 96.48 parts palmitic acid anhydride  $(C_{16}H_{31}O)_2O$ , and 100 parts of oleic acid  $C_{18}H_{34}O_2$ , to 96.81 parts of oleic acid anhydride,  $(C_{18}H_{33}O)_2O$ , no perceptible error is made by taking the content of water of the fatty acid at 3.25 per cent. Hence 3.25 per cent. has to be deducted from the weight found or, what amounts to the same thing, the per cent. number of the fatty acid has to be multiplied by 0.9675, and the remainder set down in the analysis as fatty acid. (However, in many analyses of soap this is not done, the fatty acid being set down in the analysis as hydrate, which in judging the results has to be duly taken into consideration.)

For the determination of the content of fatty acid in soap, Dr. C. Stiepel has constructed an apparatus which is intended to serve for controlling the operations in the soap factory and for calculating the cost of the soap. The apparatus is called the *soap analysator* and is shown in Fig 54. It consists of the stand, *A*, with an Erlenmeyer flask, *E*, which is held by the elastic brass clamp, *C*, and stands upon a wire net resting upon the iron ring, *B*. A tube, *a*, is fused in the flask, *E*. In the interior of the flask this tube reaches close to the bottom, and on the outside projects sufficiently to allow of a piece of rubber tube being conveniently drawn over it. Upon the ground neck of the flask sits a cylindrical tube, *F*, which is also ground in below and furnished on the lower end with two eyes by which this portion of the apparatus can, by means of a wire sling, be suspended from the scale. In addition there belong to the apparatus a small funnel with rubber tube, which rests in the ring, *D*, a short glass tube, *b*, drawn out in front and bent, and another glass tube, *c*, with a cork stopper. Heating is effected by means of the alcohol lamp, *H*. Determination of the fatty acid is effected as follows: The flask, *E*, is first tared upon a suitable scale and exactly 25 grammes of the soap to be examined are then introduced. Hard soaps are previously cut into small narrow strips, soft soaps are introduced with the assist-

ance of a glass rod, and powdered soap preparations by means of a spatula or a spoon. About 100 cubic centimeters of about 10 per cent. sulphuric acid are then poured into the flask; the latter is placed upon the wire net of the ring, *B*, secured in the clamp, *C*, and heated by means of the alcohol flame until the perfectly clear fatty acid floats upon the acid water.

FIG. 54.



When the decomposition of the soap and separation of the fatty acid have in this manner been effected, the tube, *b*, is fixed to *a* and the tube, *c*, with the stopper placed upon the flask. Now suck a few times with the mouth on *c* to draw back into the flask a drop of fat which might be in the tube *a*, or suck through *b* some warm water from a beaker into the flask whereby the drop of fat may also be brought back into

the flask. Now press off slowly through *b* the bulk of the sulphuric acid until globules of fatty acid begin to ascend in the tube. When the bulk of the sulphuric acid has thus been removed, suck through *b* about 150 ccm. of water into the flask, heat somewhat, and wash out the fatty acid by shaking from time to time. When this operation is finished remove the tubes *b* and *c*, connect the rubber tube of the funnel *G* with tube *a*, and place the upper edge of the funnel at such a height that it is slightly below the upper edge of the cylinder *F* after it has been placed in position; this cylinder should previously be weighed. The running over of the cylinder is thus prevented. Now, through the funnel *G*, bring more warm water into the flask *E* until all the fatty acid is pressed into the cylinder *F*. Then allow the apparatus to rest until the fatty acid in the cylinder is congealed. If the nature of the fatty acids, as, for instance, is frequently the case with soft soaps, be such that they do not congeal at the ordinary temperature of a room, add 5 grammes of paraffin or stearin before placing the cylinder in position. When the fatty acid is congealed remove the flask together with the cylinder and funnel from the stand, place it upon a table, and lay the funnel upside down in a beaker. Then raise the cylinder slightly so as to render it possible for the water in the cylinder to run off by the admission of air, all untidiness being thereby avoided. When the water has been run off, remove the cylinder, dry the inner and outer surfaces of the cake of fat by dabbing gently with filter paper, and again weigh the cylinder.

The content of fatty acid in the product under examination is found by a simple calculation. Suppose the weight of the empty cylinder was 23.67 grammes and it weighed after making the analysis with the use of 25 grammes of substance, 39.38 grammes, then the content of fatty acid in the product examined amounts to  $(39.38 - 23.67) \times 4 = 15.71 \times 4 = 64.84$  per cent. fatty acid, or  $62.84 \times 0.967 = 60.77$  per cent. fatty acid anhydride. If 5 per cent. of paraffin or stearin has been added, it has of course to be deducted before multiplying by 4.

It should be borne in mind that the content of fatty acid is readily found too low if, as is the case with the fatty acids of cocoanut and palmkernel oils, volatile fatty acids are present, because volatilization of the latter takes place on drying at a higher temperature. The above-mentioned fatty acids also contain water-soluble fatty acids which remain dissolved in the acid solution of alkaline salts. If the fatty acid mixture contains non-saturated fatty acids they may be oxidized in drying.

The result of the determination of the content of fatty acid in soaps made from cocoanut oil or palmkernel oil, or from stocks containing these fats is, as previously mentioned, too low, because by drying the separated fatty acids at 212° F., a considerable portion of the fatty acids of these oils volatilizes. Hefelmann and Steiner have, for instance, shown that in the case of cocoanut oil soap with a content of 65 per cent. fatty acid, only a content of 43½ per cent. was found after drying the fatty acids for about four hours at 212° F., the acid number having at the same time been reduced from 257.2 to 205.6, which proves that nearly all the fatty acids of lower molecular weight volatilize at 212° F. The above-mentioned chemists therefore propose that the potash soaps be weighed instead of the fatty acids. Franz Goldschmidt proceeds in a different way: From a large piece of soap the fatty acid is in the usual manner separated and washed. The isolated fatty acid is mixed with anhydrous sodium sulphate, which has proved to be an excellent drying agent, and allowed to stand overnight, at the ordinary temperature of a room, because at a temperature of above 97° F., the sulphate can no longer retain the absorbed water, and yields it again. Hence, if the fatty acids are solid at the ordinary temperature recourse must be had to dissolving them in ether and drying the ethereal solution with sodium sulphate. After standing for a sufficient length of time the fatty acid or its ethereal solution is filtered through a dry filter, the ether in the latter case being evaporated on a water bath at from 113° to 122° F., the lower fatty acids not

volatilizing at this temperature. Thus, without heating, we have an actually dry unchanged fatty acid, whose acid number has not been reduced by the volatilization of fatty acids of lower molecular weights. From 2 grammes of the fatty acid thus prepared the acid number is determined in the usual manner. For the actual analysis the soap is dissolved in water, decomposed in the separating funnel, and the fatty acid shaken out with ether. After washing twice with water, the ether solution is brought into an Erlenmeyer flask, rinsed with an equal volume of alcohol, and then titrated with semi-normal aqueous lye. Since by a preparatory experiment it is known how much alkali corresponds to one gramme of fatty acid, the titration directly corresponds to the content of fatty acid in the soap. It is advisable to determine by a parallel experiment the consumption of alkali of the alcohol-ether used as solvent, and at the actual titration to deduct the volume of alkaline lye consumed for the neutralization of the solvent.

To determine whether a soap contains non-saponified fat mix the finely pulverized sample of soap with sand, and after drying at 212° F. extract it with petroleum-ether. The residue remaining after the evaporation of the petroleum-ether may consist of neutral fat or hydrocarbons. The presence of the latter might be due to the fats used in the preparation of the soap containing mineral oil, or hydrocarbons having been mixed with the soap. By testing the residue as to whether it is saponifiable or not, its nature is readily established. The statement which is frequently made that the presence of free fat is indicated by the unctuous feel of the soap cannot be relied upon, since soap in the preparation of which cotton-seed oil has been used, shows also an unctuous feel.

The question *what kinds of fats have been used in the preparation of the soap* is very difficult and frequently impossible to answer by the chemist, the only guides being the determination of the melting points, equivalents of saponification and iodine numbers of the separated fatty acids. The determination of the fatty acid with the assistance of wax or stearin be-

ing not available for this purpose, another sample of the soap has to be decomposed by means of acid and the melting point, equivalent of saponification and iodine number of the resulting fatty acid determined by the methods previously given.

*Determination of rosin.*—The presence of rosin in a soap is, as a rule, readily recognized by the color and odor of the soap. The resinous acids can with certainty be established by the test of Liebermann-Storch. The fatty acids are separated from the soap by means of sulphuric acid, dissolved in acetic anhydride with the aid of a gentle heat, and the solution is allowed to cool. A little sulphuric acid of 1.53 specific gravity is then carefully added to the solution. In the presence of the smallest quantity of resinous acid a reddish-violet coloration appears; if the solution be warmed this coloration disappears almost immediately passing over into a yellowish-brown. Fatty acids do not give the violet coloration, but it should be borne in mind that cholesterin gives a similar color-reaction with sulphuric acid and acetic anhydride, and may be present in the mixture of fatty acids. It may be removed before separating the fatty acids by shaking the soap solution with ether.

*The quantitative determination of the rosin in a soap* has for a long time been a difficult problem to chemists. Gladding's test, which was formerly highly recommended and is based on the solubility of resinate of silver and the insolubility of the oleate of silver in ether-alcohol, is not reliable. Twitchell's test is more accurate. It depends upon the fact that by the action of hydrochloric acid gas fatty acids in alcoholic solution are converted into ethyl esters, while resinous acids under the same conditions remain unchanged. The test is executed as follows: Dissolve 2 to 3 grammes of the mixture of resinous and fatty acids in 10 times the volume of absolute alcohol. Place the flask containing the solution in ice-water, keeping the temperature at below 68° F., and introduce a moderate current of dry hydrochloric acid gas. The latter is at first

quickly absorbed ; in about 45 to 50 minutes the esters formed separate on the surface, and further absorption of hydrochloric acid gas ceases. The flask is then taken from the cooling water and allowed to stand for half an hour, when the contents are diluted with ten times their volume of water and boiled until the acid solution has become clear. The determination of the resinous acids may then be effected by gravimetric or volumetric analysis.

For determination by the gravimetric method add a little petroleum-ether to the contents of the flask, then bring them into a separating funnel, rinse the flask with petroleum-ether, draw off the acid solution, wash the layer of petroleum-ether, the volume of which should be about 50 ccm., with water, and shake with a solution of 5 grammes of caustic potash in 5 ccm. of alcohol and 50 ccm. of water. The rosin is saponified, while the soap remains in aqueous solution and complete separation of the two layers takes place. The solution of the rosin soap is then run off and, to avoid losses, the layer of petroleum-ether is repeatedly washed, first with an excess of dilute alkali solution, and finally with water. The rosin soap solution is decomposed with dilute hydrochloric acid, the resinous acids are dissolved in ether, and after distilling off the ether, dried at 212° F., and weighed.

For the determination of the rosin by the volumetric method, bring the contents of the flask into a separating funnel, mix them with about 75 ccm. of ether and shake. Then draw off the acid aqueous solution, wash the ethereal layer with water until acid reaction with litmus paper disappears and, after adding 50 ccm. of alcohol, titrate with semi-normal caustic potash solution using phenolphthalein as indicator. The fatty acid esters remain unacted upon, and the resinous acids are calculated with the adoption of an equivalent of 346.

The results of Twitchell's test are also not quite accurate, as has been shown by Lewkowitsch and others. The sources of error lie, according to D. Holde, in the partial non-etherization of the fatty acids, in the non-saponifiable constituents

of the rosin, in the solubility of rosin constituents in acid aqueous liquids, and in the constituents of rosin which cannot be titrated with dilute lyes in the cold, but are only saponifiable by boiling with stronger lyes. He has, therefore, in collaboration with J. Marcusson, worked out a new process for the quantitative determination of rosin, which is based upon a combination of the methods of Twitchell and Gladding. According to this process the greater portion of the fatty acids is first removed by being converted into esters according to Twitchell's method, and the separated resinous acids are then freed from the principal portion of the non-esterized fatty acids according to Gladding's method. The process is as follows: Weigh out enough of the soap to be examined so that the quantity contains about 5 grammes of fatty acid inclusive of rosin (weight *a*) and boil this with 50 ccm. of alcoholic potash solution for half an hour with an inverted condenser. Then evaporate the alkaline solution, take up the residue with water and decompose with hydrochloric acid. With soaps which contain no neutral fat, the quantity of soap weighed out can be directly decomposed with hydrochloric acid without previous boiling with alcoholic potash. Extract the separated fatty acids with ether, neutralize the acid solution, evaporate it to about 25 ccm., acidify with dilute hydrochloric acid and shake up with ether. Distil off the solvent from the total ethereal extracts. Dissolve the remaining fatty acids in 50 ccm. absolute alcohol and convert them into esters by introducing a moderately strong current of hydrochloric acid gas at a temperature not exceeding 50° F. Cooling has to be effected by ice-water. After one to two hours, when conversion into esters is finished; allow the flask to stand for about half an hour longer at the ordinary temperature of the room, then rinse the contents together with five times the quantity of water into a large Erlenmeyer flask and boil for about  $\frac{1}{2}$  hour with an inverted condenser. Then shake up the cooled liquid in a separating funnel first with 100 ccm. and then

several times with 50 ccm. of ethyl ether \* until no more coloring constituents are extracted, evaporate the aqueous liquid, previously neutralized with alcohol, to about 50 ccm., acidify, and shake up several times, each time with 25 ccm. of ether until the layer of ether becomes colorless, so as to extract the readily soluble constituents of the rosin. Shake up the combined ethereal extracts with about 50 ccm. potash lye (10 grammes caustic potash, 10 grammes alcohol, 100 ccm. water). Draw off together with the potash lye the brown layer which appears between the ether and the potash lye, and which is soluble in water. It contains a considerable portion of the rosin soaps which are soluble with difficulty in the lye. Next thoroughly wash † the layer of ether first with water, because the rosin soaps dissolve in it with ease, then twice with 10 ccm. potash lye and finally shake out with water until the latter remains colorless. For the removal of mechanically adhering particles of ester shake the combined aqueous and alkaline extracts with 50 ccm. ether. Lift off the layer of ether and shake it out again with 5 ccm. potash lye and combine the latter with the main portion of the alkaline extract. Now acidify the combined alkaline extracts and exhaust with about 50 ccm. ether each time. Neutralize the acid solution, evaporate it as much as possible, acidify and again extract with ether. Now combine all the ether extracts, wash with 20 ccm. water and distil off the solvent. The resinous acids thus obtained, which are, however, still contaminated with fatty acids not converted into their esters, are weighed in a tared dish after evaporating the remnants of ether on a water bath, some absolute alcohol for the removal of water being if necessary added.

For the subsequent operation about 0.4 to 0.6 gramme (weight *b*) of the acids thus obtained is dissolved in 20 ccm.

\* Dark oxyacids separating out in ether are dissolved, after running off the ether, in a little alcohol, and the resulting solution is added to the ether solution.

† In some cases, as for instance, in the presence of phytetoleic acid and much rosin, washing has to be quite frequently repeated until the water is colorless.

of 95 per cent. alcohol in a graduated flask having a capacity of 100 ccm. If a smaller quantity than this of acids has been obtained, the proportionate quantities of alcohol-ether mixture given below must be correspondingly altered. With larger quantities of acids the entire product obtained is dissolved in enough 95 per cent. alcohol that 20 ccm. of the solution, which for further examination can be withdrawn by means of a pipette, contain about 0.5 gramme of acid.

The solution prepared as above described is brought into a graduated cylinder holding 100 cubic ccm. and whilst being vigorously agitated, is mixed with a drop of phenolphthalein (if the solution is very dark 2 to 3 drops of alkaline blue 6 B are taken) and sufficient drops of concentrated aqueous soda lye (1 part NaOH, 2 parts H<sub>2</sub>O) until an alkaline reaction is just produced. The cylinder loosely closed is then for a short time heated on the water bath. The contents of the cylinder are then allowed to cool, made up with ether to 100 ccm., and shaken. One gramme of powdered and dried silver nitrate is then added and the whole shaken 15 to 20 minutes to convert the acids into silver salts. When the precipitate consisting of silver oleate has thoroughly settled, for which purpose it may be necessary to allow the mixture to stand overnight, about 70 ccm. of the fluid are brought by means of a pipette, and if necessary with the assistance of a filter, into a second graduated cylinder holding 100 ccm. This portion is thoroughly shaken up with 20 ccm. of dilute hydrochloric acid (1 part concentrated hydrochloric acid, 2 parts water) and after drawing off the layer of ether, the aqueous fluid is shaken twice more, each time with 20 ccm. of ether.

The combined ethereal extracts are now shaken with about 20 ccm. of water for the removal of the hydrochloric acid, separated from the water, filtered into a flask and freed by distillation from the greater portion of the ether. The residue, about 10 ccm., is rinsed into a weighed dish and evaporated, and is finally freed from moisture and adhering solvent by heating for a short time to from 230° to 239° F., until the liquid is just clear.

The weight  $c$  of the residue is calculated upon the total quantity of acid  $b$  used for the Gladding process, *i. e.* if 70 ccm. of alcohol-ether solution have been used for the test 100 ccm

$$d = \frac{c}{10} 100 \text{ grammes of resinous acids.}$$

The value  $d$  thus found gives either directly or by simple reduction the content of resinous acids in the quantities of acids obtained according to Twitchell, as well as in the quantity originally used for the test. From  $d$  results the percentage  $e$  of resinous acids in the substance originally used as follows:—

$$e = \frac{d}{a} 100$$

As the quantity of resinous acids  $e$  obtained still contains small quantities of fatty acid, 0.4 per cent. has to be deducted as a mean correction.

When the quantity of rosin as above determined amounts to less than 20 per cent., the non-saponifiable portion of the rosin is taken into account by the correction + 8 per cent. on the quantity of rosin found.\* The resinous acids having been found according to the foregoing  $e$ —0.4 per cent., the mean content of rosin  $f$  is calculated according to the following formula:—

$$f = \frac{100 (e - 0.4)}{92}$$

For the direct determination of the non-saponifiable substances in the presence of over 20 per cent. resinous acid, the ethereal solution of the esters previously obtained is, after complete removal of the resinous acid, saponified with 25 ccm. of normal alcoholic potash lye. The soap solution is com-

\* Holde and Marcusson found in an American rosin 8 per cent. of non-saponifiable constituents; other statements vary between 5 and 15 per cent.

pounded with 150 ccm. of water and twice extracted, each time with 150 ccm. of ether. The greater portion of the ether is distilled off at the ordinary temperature of the room, since at a higher temperature volatile substances escape. The remaining oily residue only contains small quantities of acid soap, which is removed by treatment with a little alcoholic potash, slow evaporation of the alcohol and extraction with petroleum-ether. The weight of the residue thus purified is calculated on 100 parts of the quantity of substance used and added to the amount of resinous acids e—0.4 found.

For the determination of rosin in soaps which contain non-saponifiable oils the quantity of sample to be weighed out is so adjusted that the weight of the fatty acid to be subsequently separated amounts to about 5 grammes. The sample is saponified with the addition of benzol free from thiophene, and from the soap solution thus obtained the non-saponifiable substances are extracted by the process of Spitz and Hoenig. The soap solution is brought into a separating funnel, the flask which contained it, rinsed first with 50 per cent. alcohol and next with 50 ccm. of petroleum-ether (boiling point up to 122° F.). The contents of the separating funnel are vigorously shaken and then allowed to rest; the petroleum-ether separates quickly and sharply from the alcoholic soap solution. The latter is drawn off, the petroleum-ether washed twice or three times with 50 per cent. alcohol, each time with 10 to 15 ccm., and the alcoholic washings are combined with the original soap solutions. Extraction of the soap solution with petroleum-ether is repeated until the ether leaves no grease spot upon paper. For the removal of the small quantities of soap taken up, each of the petroleum-ether extracts is washed with 50 per cent. alcohol. In most cases three extractions with petroleum-ether suffice; the remaining solution of rosin and fatty soaps are further treated as previously described. Accurate determination by the gravimetric method of the non-saponifiable constituents of rosin besides the non-saponifiable oils being in this case not always possible, Holde and Marcusson add the

mean value of 8 per cent. to the quantity of resinous acid found.

*Determination of alkalies.*—The alkali metal of a soap is either potassium or sodium or a mixture of both. To determine the kind of alkali, separate the fatty acids from a solution of soap and test with alcoholic solution of chloride of platinum which, in the presence of potassium, gives a yellow precipitate. The quantitative determination of the alkalies, if only one be present, is effected by the alkalimetric method. Decompose the soap with an excess of normal acid, and after allowing the fatty acids to separate, determine the quantity of acid added in excess by titrating with caustic alkali. The difference gives the quantity of acid used for the neutralization of the alkali of the soap and from it the quantity of the alkali itself is found. If the decomposition of the soap is not readily effected by the acid, add some spirit of wine, which dissolves the soap and facilitates its decomposition. Heating or boiling should by all means be omitted. Dissolve, for instance, 25 grammes of grained soap in hot water, add 55 ccm. of normal acid, heat to separate the fatty acid, and allow to cool. After cooling and filtering wash the fatty acids remaining upon the filter with hot water.

The filtrate is titrated back with normal soda lye, methyl-orange being approximately used as indicator. Suppose 4 ccm. of soda lye have been required for this purpose, then for fixing the alkali contained in 25 grammes of soap 51 ccm. normal acid have been used, hence for that in 100 grammes of soap 204 ccm. Since 1 ccm. normal acid corresponds to 0.031 sodium oxide, 100 grammes of soap contained  $204 \times 0.031 = 6.324 \text{ Na}_2\text{O}$ .

With hard soaps the alkali is generally calculated to sodium oxide  $\text{Na}_2\text{O}$ , and with soft soaps to potassium oxide  $\text{K}_2\text{O}$ , no attention being paid to the fact that hard soaps occasionally also contain potassium, and soft soaps, in most cases, sodium. If the quantity of both of the alkalies is to be found, the total alkali in a sample is first alkalimetrically determined, while

another sample is decomposed with hydrochloric acid and the potassium in the solution determined with platinum chloride. From the potassium found and the total quantity of alkali, the sodium is then calculated.

*Whether a soap contains free alkali* is recognized by adding to its alcoholic solution a small quantity of phenolphthalein which produces a red coloration; further by dropping upon the fresh cut surface of the soap a small quantity of mercuric chloride which produces a yellow coloration, or a small quantity of mercurous nitrate, which gives a black coloration. The test with mercuric chloride is however, not always reliable, as the reaction does not appear when the soap, in addition to a small content of caustic alkali or alkaline carbonate, contains potassium chloride or sodium chloride. In the case of a dry soap dabbing a freshly-cut surface of it with alcoholic phenolphthalein solution produces a red coloration only when the soap contains free caustic soda; if the soap is moist, the red coloration may also be caused by alkaline carbonate, silicate or borate.

*To determine the quantity of non-saponified alkali* dissolve 5 to 10 grammes of soap in alcohol free from acid, filter and rinse thoroughly with alcohol. Titrate the filtrate with  $\frac{1}{10}$  normal sulphuric acid, using phenolphthalein as indicator, until discolouration takes place, and thus find the content of free caustic soda. Wash the residue collected upon the filter several times with water, titrate the solution with  $\frac{1}{10}$  normal acid using methyl orange as indicator, and thus determine the quantity of alkaline carbonate.

*Determination of filling materials.*—The filling materials used for soap are partially salts soluble in water, especially potassium chloride, sodium chloride, potassium sulphate, sodium sulphate, alkaline carbonates and silicate of soda, and partially mineral substances insoluble in water, such as talc, heavy spar, infusorial earth, etc., and organic substances, such as starch. To determine admixtures dissolve the soap cut into fine shavings in 8 to 10 times its quantity of 90 per cent.

alcohol by moderate heating on a water bath. Then filter the solution and after washing the residue with alcohol and drying at 212° F., weigh it. Only transparent soaps prepared with the assistance of spirit are soluble in alcohol without leaving a residue.

The residue left behind by filled soaps is very considerable. It may contain, 1. Salts soluble in water, especially the chlorides, sulphates and carbonates of the alkalies, further silicate of soda, borax, etc. 2. Mineral substances insoluble in water, such as chalc, clay, lime, infusorial earth, etc. 3. Organic substances such as starch, glue, mucilage.

*To determine alkaline carbonate, silicate, and borate,* extract the residue with cold water. Determine in one portion of the filtered solution the alkali fixed on carbonic acid, silicic acid and boric acid by titration with hydrochloric acid, using methyl orange as indicator. After acidifying the liquid with hydrochloric acid any silicic acid derived from silicate of soda which may be present can also be separated by evaporation. The filtrate obtained after the separation of the silicic acid may be used for testing for boric acid by moistening with it a strip of turmeric paper and drying at a moderate heat. The aqueous solution of boric acid possesses the characteristic property, even when acidified with hydrochloric acid, of producing on drying on turmeric paper a brown red spot which, when touched with ammonia, changes to black-blue.

When the residue of the soap contains borax or silicate of soda besides soda, a portion of it may be used for ascertaining the amount of soda by determining the carbonic acid.

*Silicate of soda* may also be determined, though somewhat less accurately, by dissolving the soap in water and decomposing it with an acid. The fatty acids float on the top, while the silicic acid sinks to the bottom or remains suspended in the fluid. It is collected by filtering, washed, dried, ignited and weighed and, according to Waltke, calculated into silicate  $\text{Na}_2\text{Si}_4\text{O}_9$ .

Suppose that in this manner in 5 grammes soap powder 0.5

gramme silica ( $\text{Si}_2\text{O}_5$ ) has been found, then 100 grammes of the powder contain 10 grammes  $\text{SiO}_2$ . Since the equivalent weight of  $\text{SiO}_2 = 60$ , that of  $4\text{SiO} = 240$ , and of  $\text{Na}_2\text{Si}_4\text{O}_9 = 302$ , the content of silicate of soda is found from the determined  $\text{SiO}_2$  by the formula :  $302 = 10 : x$ ,  $x = 12.5$  per cent. When sodium carbonate, sodium silicate and sodium borate are present side by side, the quantitative determination of the three combinations may, according to Waltke, be performed as follows : The portion of 3 to 5 grammes of the sample which is insoluble in alcohol, is dried and weighed until its weight remains constant. With a portion of the residue a direct determination of carbonic acid is made and in the remainder the silicic acid is separated by evaporation with hydrochloric acid. The filtrate from the latter is used for the determination of the total sodium as sodium chloride or sodium sulphate. The quantity of sodium corresponding to the sodium carbonate and silicate is deducted from the total quantity of sodium, and the balance of sodium calculated into sodium borate. If, besides the three salts above mentioned, sodium chloride and sodium sulphate are also present, the quantities of these must also be taken into account in calculating the sodium borate.

To ascertain the presence of sodium chloride and Glauber's salt, chlorine and sulphuric acid are determined in portions of the aqueous extract of the residue of the soap insoluble in alcohol by precipitating with silver nitrate and barium chloride respectively. The content of chlorine, and consequently also that of sodium chloride can, according to Horn, be determined with far greater accuracy by dissolving the soap in water, separating the fatty acid with dilute nitric acid and precipitating the filtrate with silver nitrate. For determining the sulphuric acid, Horn incinerates the soap, extracts the ash with hydrochloric acid, and precipitates with barium chloride.

The portion of the residue insoluble in water is ignited to destroy the organic substances, and the ash may then be further examined both qualitatively and quantitatively.

Regarding the *determination of organic substance*, cold water extracts the *dextrin* from the portion insoluble in alcohol, and it can be again precipitated from the aqueous solution with alcohol. By effecting precipitation in a beaker weighed together with a glass rod, and stirring vigorously, the dextrin deposits on the sides of the beaker. Pour off the fluid, wash with alcohol, dry the beaker with the dextrin at 212° F., and weigh.

The presence of starch (*potato flour*) in the residue insoluble in alcohol is recognized by means of the microscope and by the blue coloration produced with iodine solution. If starch is present, dry the residue after the extraction with cold water at 212° F., weigh, incinerate, and weigh again. The difference gives approximately the content of organic substances and, in the case of others not being present, that of starch. Or, to convert the starch into sugar, boil the residue with dilute sulphuric acid, constantly replacing the water lost by evaporation, neutralize the solution with barium carbonate, filter, and determine the sugar by titrating with Fehling's solution.\*

According to Dr. Karl Bauer, 10 grammes of the soap are dissolved in a porcelain dish with the assistance of heat in 50 ccm. of water, the fatty acids separated by the addition of acid, and after cooling the cake of fat is lifted out and carefully cleaned by squirting with water. Add to the aqueous residue 0.5 ccm. of pure concentrated sulphuric acid and boil for about one hour. The water lost by evaporation is from time to time replaced, and after adding 0.5 ccm. more of sulphuric acid, boiling is continued for 1 to 1½ hours longer. By boiling with the acid the starch is converted into sugar, which may

\* Fehling's solution is prepared by dissolving, on the one hand, 34.639 grammes of crystallized cupric sulphate in water, and diluting the solution to 500 ccm. and, on the other hand, dissolving 173 grammes sodium potassium tartrate (Rochelle salt) and 50 grammes caustic soda to make 500 ccm. For use mix equal parts of the solutions. 1 ccm. of the solution ready for use corresponds to 0.005 sugar.

be determined by titrating with Fehling's solution. Neutralize the fluid with potash lye and bring it up to exactly 200 ccm. Heat 50 ccm. of this fluid in a dish to the boiling point and run in from a burette, Fehling's solution, which will produce a red precipitate. Allow to cool and test the supernatant fluid for copper. This is done by bringing by means of a glass rod a drop of the supernatant fluid upon a porcelain plate and carefully adding a drop of potassium ferrocyanide solution (1 : 20) acidified with acetic acid. On the point of contact of the two drops a zone of only a very slight red brown color should be formed. The remaining 150 ccm. are used for controlling titrations.

If, for instance, 11.12 grammes of soap have been taken for the test and for 50 ccm. of the sacchariferous fluid 7.1 ccm. of Fehling's solution have been used, hence for 200 ccm. 28.4 ccm., then, since 1 ccm. corresponds to 0.005 sugar,  $28.4 \times 0.005 = 0.142$  sugar. For calculating to starch the number has to be multiplied by 0.9; we have thus,  $0.142 \times 0.9 = 0.1278$  starch. Since potato flour contains about 20 per cent. foreign substances, such as ash, nitrogenous substances, and moisture, Braun adds the fifth part of the number found, the result being, therefore,  $0.1278 + 0.0256 = 0.1534$ , and according to the proportion :  $11.12 : 0.1534 = 100 : x$ ,  $x = 1.378$  per cent. starch in the soap.

*To determine the presence of glue*, the portion of the soap insoluble in alcohol is extracted with hot water. The solution will gelatinize on cooling and gives no precipitate with tannic acid.

*The presence of petroleum* is ascertained by moderately heating the soap on a water bath or on a sand bath, whereby the petroleum distils off. When a sufficient quantity of soap has been subjected to distillation, the distillate can be measured, or, after separating the water carried over with it, measured.

*Determination of glycerin.* Dissolve about 25 grammes of the soap to be examined in hot water, and compound the solution with dilute sulphuric acid until acid reaction takes place.

Then melt the fatty acids together with wax, and after cooling, lift off the cake of fat, accurately neutralize the fluid with sodium carbonate and evaporate it to dryness on a water bath. Treat the residue which consists of sodium sulphate and glycerin with alcohol, whereby the sodium sulphate remains undissolved behind. Filter the alcoholic solution, then evaporate, again treat the residue with alcohol, again filter the solution and evaporate it in a platinum dish on a water bath. Great accuracy cannot be claimed for this method on account of the volatility of the glycerin. For a more accurate determination of the glycerin, dissolve, according to the suspected content of glycerin, 1 to 10 grammes of the soap in water, or, if organic substances insoluble in water be present, in methyl alcohol. Filter the solution, and after evaporating the methyl alcohol, if used, separate the fatty acid, and then proceed with the acid filtrate in the same manner as for the determination of glycerin in fats.

Grained soaps contain no glycerin, or only a very inconsiderable quantity of it; soaps prepared by the cold process contain about 5 per cent. If a soft soap contains no glycerin it is an indication that it has been boiled from fatty acids.

*Determination of sugar.*—Sugar in considerable quantities is contained in cheap transparent soaps. The quantitative determination of *cane sugar* may be effected either by inversion with Fehling's solution, or polarimetrically. For the first process the hot aqueous solution of a weighed quantity of the sample is heated with as small an excess as possible of  $\frac{1}{2}$  normal sulphuric acid, the fatty acids are separated, the aqueous solution is neutralized and concentrated to about 75 ccm. This solution is in the usual way inverted with hydrochloric acid, and the inverted sugar gravimetrically determined with Fehling's solution.

Polarimetrically, Wilson proceeds as follows: 10 grammes of soap are dissolved in 150 ccm. of water at 112° F., and saturated solution of magnesium sulphate in small excess is added drop by drop whilst constantly stirring. The mag-

nesium soap is then filtered off through a purified filter, and washed with hot water containing magnesium sulphate. The filtrate which is generally somewhat alkaline is nearly neutralized with dilute nitric acid, evaporated to about 40 ccm., allowed to cool, slightly acidified with a few drops of dilute nitric acid, clarified, as usual, with lead acetate and, after filtering polarized.

In the presence of both glycerin and cane sugar Donath and Mayrhofer recommended to add to the solution containing both substances, slaked lime for the formation of calcium saccharate and ignited sand, then to evaporate and extract the residue with a mixture of equal volumes of alcohol and ether. The solution contains all the glycerin.

*Determination of alcohol.*—Transparent soaps being, at the present time, prepared with alcohol as well as without, it may sometimes be important to know whether a soap contains alcohol. For its determination, mix, according to Valenta, 50 to 60 grammes of the soap to be examined with pumice stone, and distil on a paraffin-bath, first at 230° F. and then at 248° F. With the distillate the iodoform test is made, which is executed, according to Hager, as follows: The fluid to be tested is compounded with 5 to 6 cubic centimetres of a 10 per cent. potash solution, and after heating to from 104° to 122° F., 16 to 20 per cent. potassium iodide solution saturated with iodine is added until the fluid assumes a yellow-brownish color. If the color does not appear on shaking, add by means of a glass-rod just sufficient potash lye entirely to decolorize the fluid. Immediately, or after standing a short time, yellow crystals of iodoform are separated out, which, when viewed under the microscope, present a star-like appearance or one of hexagonal tablets.

*Determination of essential oils.*—The separation of essential oils used for perfuming can, according to Barfoed, be effected by two methods: Extract the soap at an ordinary temperature with ether, filter through a filter moistened with ether, and rinse off with the same liquid. Shake the solution with water

to remove any soap which may have passed into it, and then evaporate. *Or*, dissolve the soap in water, add to the solution a small quantity of sulphuric acid to prevent strong foaming, and distil off the essential oil with steam. From the distillate the oil is collected by shaking with ether. The nature of the oil is recognized by the odor.

For the quantitative determination of *turpentine*, the soap, according to Dr. Karl Braun, is dissolved in water in a flask, saturated with sulphuric acid and distilled for about one hour. The distillate is caught in a graduated cylinder and the quantity of turpentine which collects over the water, read off.

*Determination of carbolic acid.*—The determination of carbolic acid (phenol and cresol) is, according to Lewkowitsch, made as follows: Weigh out a larger quantity of the sample, about 100 grammes; dissolve it in hot water, and add enough soda lye for the solution to become strongly alkaline. Next separate the soap by the addition of common salt, filter off the grain, and wash the soap with common salt solution. The solution, which now contains the phenol and cresol as sodium salts, is evaporated and any dissolved soap precipitated by again adding common salt. Now filter again, evaporate to a small quantity, bring the solution into a graduated cylinder holding from 50 to 100 ccm., add enough common salt that some of it remains dissolved and acidify with sulphuric acid. The volumes of the separated phenols are read off, the number of cubic centimeters being taken into account as so many grammes.

*Determination of ammonia.*—According to Dr. Karl Braun, about 5 grammes of soap are accurately weighed out and brought into a flask holding  $1\frac{1}{2}$  liters. Soft soaps are weighed upon a piece of paper previously weighed, and dissolved in about 50 ccm. of water. The flask is connected with a Liebig condenser placed obliquely. The condenser is furnished with a connecting pipe fitting air-tight, which projects into a receiver containing 100 ccm.  $\frac{1}{6}$  normal hydrochloric acid. A large beaker may serve as receiver. When the apparatus has

been put together, strong soda lye or potash lye is added to the soap solution in the flask until the fluid reacts strongly alkaline and the soap floats as grain on top. After ascertaining that all the corks are tight, the flask is heated for about 1½ hours. The stronger soda lye drives out the weaker ammonia. The latter is immediately fixed by the hydrochloric acid in the receiver, ammonia chloride being formed. An indicator is added to the hydrochloric acid in the receiver, cochineal tincture\* being very suitable for this purpose. If the red coloration should change to violet, add about 50 ccm. of hydrochloric acid. After boiling for 1½ hours it may be supposed that all the ammonia has been carried over. Now carefully detach from the condenser the connecting pipe that projects into the receiver and test with red litmus paper as to whether the distillate still reacts alkaline. If such be the case, distillation must be continued; if the litmus paper remains unchanged, the flame under the flask is removed. Care must be taken to see that bubbles of foam are not carried over, as otherwise the determination becomes inaccurate. The connecting pipe, after it has been detached, is rinsed with water and the distillate is allowed to cool. Then titrate with  $\frac{1}{10}$  normal potash lye until the appearance of a violet coloration and note the number of cubic centimeters used.

Suppose 5.67 grammes of substance have been used,

In the receiver,	100 ccm.	$\frac{1}{10}$ HCl,
Titrated back,	17 ccm.	$\frac{1}{10}$ KOH,
Used,	83 ccm.	$\frac{1}{10}$ HCl.

\* Since 1 ccm.  $\frac{1}{10}$  HCl = 0.0017 NH<sub>3</sub>, 83 ccm. HCl = 0.0017  $\times$  83 = 0.1411 NH<sub>3</sub>. Hence the content of ammonia in percents results from the formula :

\* Cochineal tincture is prepared by pouring 30 grammes of alcohol and 70 grammes of water over 10 grammes of cochineal. Heat to about 122° F., allow to stand several days to settle, and filter.

$5.67 : 0.1411 = 100 : x$  hence  $x = 2.48$  per cent. ammonia ( $\text{NH}_3$ ),  
or calculated upon ammonia chloride, since 1 ccm.  $\frac{1}{10}$  HCl = 0.00535  $\text{NH}_4\text{Cl}$ ,  $x = 7.83$  per cent. ammonium chloride ( $\text{NH}_4\text{Cl}$ ).

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